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Materials Research Activities at the National Bureau of Standards (1975-1982) Pertaining to Active Solar Heating and Cooling Systems

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
National Engineering Laboratory
Center for Building Technology
Building Materials Division
Washington, DC 20234

November 1983

Prepared for
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Office of Solar Heat Technologies
Active Solar Heating and Cooling Division
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MATERIALS RESEARCH ACTIVITIES AT THE NATIONAL BUREAU OF STANDARDS (1975-1982) PERTAINING TO ACTIVE SOLAR HEATING AND COOLING SYSTEMS

Charles W. C. Yancey

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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, *Secretary*
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director*

ABSTRACT

A summary of the solar energy materials-related research projects conducted by the National Bureau of Standards, for the U.S. Department of Energy, since 1975 is presented. Research studies concerned with materials that are utilized in the collector, transport and storage subsystems are summarized. Materials research areas covered by the documentation include: cover plates, absorber coatings,thermal insulation, sealants, containment materials, heat transfer fluids, hoses and storage media materials. The primary objectives, scope and principal results of the various studies are presented. The relationship between test results and subsequent consensus standard adoption or revision is drawn where applicable.

Key words: absorber coatings; containment materials; cover plates; durability; heat transfer fluids; hoses; performance tests; sealants; solar collectors; solar energy storage subsystem; storage media; thermal insulation; transport subsystem.

TABLE OF CONTENTS

		Page
CHAPTER 1.	INTRODUCTION	1
	1.1 BACKGROUND	1
	1.2 ORGANIZATION OF THE REPORT	2
CHAPTER 2.	COLLECTORS	4
	2.1 COVER PLATES	4
	2.1.1 Introduction	4
	2.1.2 Optical and Mechanical Property Measurement and Durability Assessment	4
	2.1.3 Parametric Study of Optical Properties	7
	2.1.4 Weathering Performance Measurement	13
	2.1.5 Hail Impact Testing	18
	2.2 ABSORBER PLATES	21
	2.2.1 Introduction	21
	2.2.2 Laboratory and Field Experimentation	22
	2.2.3 Parametric Study of Optical Properties	26
	2.2.4 Analytical Modeling	28
	2.2.5 Laboratory Evaluation Devices	32
	2.2.6 Evaluation of Selective Coating Candidates	33
	2.3 INSULATION	35
	2.3.1 Introduction	35
	2.3.2 Research Activities	35
	2.4 SEALANTS AND GASKETS	39
	2.4.1 Introduction	39
	2.4.2 Research Activities	39
CHAPTER 3.	TRANSPORT AND STORAGE SYSTEM COMPONENTS	43
	3.1 CONTAINMENT MATERIALS AND HEAT TRANSFER FLUIDS	43
	3.1.1 Introduction	43
	3.1.2 Screening Test for Metal/Heat Transfer Fluid Combinations	43
	3.1.3 Screening Test for Plastic Materials/Heat Transfer Fluid Combinations	44
	3.1.4 Simulated Service Testing for Corrosion	46
	3.2 HOSES AND FLEXIBLE COUPLINGS	53
	3.2.1 Introduction	53
	3.2.2 Research on Rubber Hoses	53

TABLE OF CONTENTS (continued)

		Page
3.3	STORAGE MEDIA	55
3.3.1	Introduction	55
3.3.2	Background	55
3.3.3	Problem Identification	58
3.3.4	Performance Test Method Needs	58
CHAPTER 4.	SUMMARY OF ACCOMPLISHMENTS AND CURRENT STUDIES	59
4.1	RESEARCH ACCOMPLISHMENTS	59
4.2	FISCAL YEAR 1982 RESEARCH ACTIVITIES AT NATIONAL LABORATORIES	59
CHAPTER 5.	ACKNOWLEDGEMENTS	65
CHAPTER 6.	INDEX OF REFERENCES	66

1. INTRODUCTION

1.1 BACKGROUND

In 1974, the U.S. Department of Energy (DoE) initiated the Active Solar Heating and Cooling (ASHC) Program to foster the advancement of solar energy technology and the emergence of the solar energy industry. This report summarizes the solar energy-materials-related research projects that were conducted since 1975 by the National Bureau of Standards (NBS) under the auspices of the U.S. Department of Energy's (DoE) Active Solar Heating and Cooling (ASHC) Program. Some of the research conducted by other national laboratories (i.e., Los Alamos National Laboratory (LANL) and Argonne National Laboratory (ANL)) under the ASHC Program is also documented here. The reader is referred to companion documentation reports by those laboratories for a complete listing of their activities. Research projects and field studies conducted by universities and private organizations for the DoE ASHC Program are not documented herein.

The primary objective of most of the research described herein was to obtain initial performance and long-term durability data which are needed for the development of performance criteria and evaluation standards for materials used in solar collectors or in transport and storage system components. Much of the background covering the development of performance criteria and evaluation-type test methods can be found in a NBS report entitled "Plan for the Development and Implementation of Standards for Solar Heating and Cooling Applications" [1].* The report contains the results of a DoE-sponsored study which sought to identify high priority standards needs and, through their development, to stimulate the use of solar energy heating and cooling systems in buildings. In reference [1], the NBS, working in conjunction with the American National Standards Institute (ANSI) Solar Energy Steering Committee, tabulated some 210 standards which needed development. These standards were ranked in descending order of priority.

The ranking of the materials test methods and specifications listed in reference [1] was largely corroborated by the findings of a survey and field study conducted by the NBS [2]. By identifying and documenting materials-performance problems in operating solar energy systems, the investigators sought to: 1) compile a data base on materials performance, 2) identify materials which need performance tests, 3) identify and assess existing standards that may be applicable to solar materials evaluation and 4) provide recommendations for the development of needed performance standards. The materials standards needs and their relative priority rankings are listed in table 1.1 as they were presented in reference [2]. The "key" properties and tests associated with each of the standards areas were also identified.

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* Figures in brackets pertain to the references listed in the index located at the end of the report.

1.2 ORGANIZATION OF THE REPORT

The summaries that follow are intended to present the primary objectives, the scope and the principal results of the various studies conducted by NBS. When a consensus standard has been adopted or an existing standard has been subsequently revised, that action is so indicated. The details of the affected standard are not presented here as they are discussed in another NBS report, "Test Method Development and Research for Evaluating Active Solar Energy Systems, Materials and Components." A description of the individual materials and material combinations are presented and test variables are noted. The information has been organized according to the major solar energy subsystems: Collector, Transport, and Storage.

Research studies concerned with the materials used in components of the collector subsystem are summarized in chapter 2. Included in that chapter are sections on cover plates, absorber coatings, insulation, and sealants and gaskets. Chapter 3 contains sections on research activities relating to materials utilized in either the transport or storage subsystems (containment materials, heat transfer fluids, hoses, and fluid/storage media). Remaining high-priority research needs, and some of the current activities sponsored by DoE aimed at fulfilling those needs, are described in chapter 4.

Units of measurement used in this summary are the same as those provided in the reports summarized.

Table 1.1 Recommended Priorities for the Development of Materials-Related Standards (from Reference [1])

Materials Standards Area	Priority
Absorptive coatings	High
Collector insulation	High
Cover plates	High
Heat transport liquids	High
Metallic containment	High
Nonmetallic containment and absorber substrates	High
Seals	High
Stagnation testing of collector subsystems	High
Surface temperature measurements	High
Flexible couplings	Medium
Reflective surfaces	Medium
Thermal storage media	Medium
Transport and storage insulation	Medium
Coatings/liners for transport or storage subsystems	Low
Desiccants	Low
Dielectric insulators	Low
Filters/getters	Low

2. COLLECTORS

2.1 COVER PLATES

2.1.1 Introduction

In a flat-plate solar collector, the cover plate has at least four functions which affect collector performance: 1) the cover plate should permit the maximum possible transmission of solar energy to the absorber plates; 2) the cover should minimize the reflection of infrared solar radiation from the absorber plate back into the atmosphere; 3) the cover plate should minimize convective heat losses from the absorber plate by maintaining a confined air space between the surface and the absorber; and 4) the cover plate should protect the interior of the collector from potentially harmful external weather conditions. Cover plate materials should be selected on the basis of their optical, thermal and physical performance as well as their durability and cost. Several research studies have sought to establish a technical basis for the development of standard test methods for cover plate materials, that can be used for the evaluation of their optical and mechanical properties and in assessing the long-term durability of candidate materials. In the paragraphs that follow, the details of those studies conducted by NBS are summarized.

2.1.2 Optical and Mechanical Property Measurement and Durability Assessment

During 1976 and 1977, the National Bureau of Standards performed laboratory and field studies [3] on 10 cover plate materials to measure critical optical and mechanical properties and to assess material durability under exposure to natural and simulated weathering. The materials used in the study were typical of those commercially available in 1976 and are listed in table 2.1. The thicknesses of the specimens were representative of those used in solar collectors. The two glass sheet materials differed in iron oxide content which directly affects the solar energy transmittance. The two fiber reinforced plastics were obtained from different manufacturers and contained different resins. The environmental degradation factors studied were heat aging and solar radiation. Three procedures which simulated these factors were developed. The three aging procedures were: 1) heat stability exposure in a mechanical convection oven, 2) artificial solar radiation exposure in a Xenon arc weathering machine, and 3) natural weathering exposure on minicollector boxes. The latter two procedures were used to permit comparison of the exposures and to determine whether the effects on the materials were independent or synergistic. The material properties measured were: 1) solar energy transmittance, 2) linear dimensional changes, 3) warpage, and 4) the effects of dirt retention. The first two properties were measured by ASTM standard procedures while the latter two were measured by procedures developed during the study. Solar energy transmittance was measured by both methods A and B of ASTM Standard E424-71 [4]. Method A is performed in the laboratory utilizing an integrating sphere spectrophotometer. Method B is conducted outdoors using a pyranometer in an enclosure and the sun as the energy source.

Table 2.1 Cover Plate Materials Tested for Physical Properties and Durability (from reference [3])

Material	Nominal Thickness mm	(in)
Glass (0.01% iron oxide)	3.2	(1/8)
Glass (0.10% iron oxide)	3.2	(1/8)
Fluorinated (ethylene propylene) copolymer	0.025	(0.001)
Poly(vinyl fluoride)	0.10	(0.004)
Poly(ethylene terephthalate)	0.13	(0.005)
Acrylic	0.076	(0.003)
Poly(methyl methacrylate)	1.5	(0.06)
Polycarbonate	1.02	(0.04)
Glass fiber reinforced plastic	1.02	(0.04)
Glass fiber reinforced plastic	1.5	(0.06)

Temperatures for the heat aging tests were based upon maximum cover plate stagnation temperature measurements obtained during a preliminary study. Test temperatures were set at 75°C (167°F), 100°C (212°F), 125°C (257°F), and 150°C (302°F). The test specimens were suspended in an oven described in ASTM Standard D794, and exposed to the test temperatures for time intervals ranging up to 2,000 hours. Once the specimens were removed from the oven, measurements were made of solar transmittance, warpage and linear dimensions, and they were then discarded. In the artificial solar exposure tests, a xenon arc light apparatus was used to simulate the spectral distribution of sunlight. Test specimens were exposed to the artificial radiation according to the procedure described in ASTM Standard D2565-79. The specimens were removed for evaluation at time intervals from 250 to 4,000 hours. For the natural weathering exposure tests, special "minicollectors" containing the cover plate specimens were assembled to obtain the elevated temperature levels associated with stagnation conditions. The minicollectors were weathered at three sites: New River, Arizona; Miami, Florida; and Gaithersburg, Maryland. The weathering racks were exposed according to ASTM Standard D1435-75. Sets of weathering specimens were removed for property testing at 3, 6, 12, 18, and 24 month intervals. (In a continuation of the weathering exposure tests, some specimens were left at the sites for 36 or 48 months before being removed for evaluation. A discussion of the results of the extended exposure testing is presented in section 2.1.4.)

Most of the data were presented in terms of either percent of solar energy transmittance versus time of exposure or spectral transmittance (percent transmittance versus wavelength). In addition, data indicating the percent change in linear dimensions and the amount of warpage after heat aging exposure were presented. There were a number of conclusions drawn as a result of the laboratory and field studies; some of them are enumerated below:

1. Method A (spectrophotometer) of ASTM E424 has better precision and repeatability than Method B (outdoor) and is therefore preferrable to Method B for determining solar energy transmittance of cover plate materials.

2. Accelerated laboratory aging procedures, such as those performed in the study offer an effective means of screening candidate cover plate materials.

3. Heat aging at elevated temperature (150°C (302°F)) for a relatively short time (500 hours) is an effective means of identifying materials which will develop problems in outdoor weathering.

4. Measurement of linear dimensions after 10 hours exposure at a specific temperature is sufficient to establish trends and determine approximate dimensional changes due to that specific temperature.

5. Artificial weathering with a xenon arc light is helpful in distinguishing materials that are affected by solar radiation.

6. The minicollectors adequately simulated operating and in some cases stagnation temperatures obtained by flat plate collectors.

7. Dirt and dust retention is a major cause of solar energy transmittance decrease in all cover plate materials. Solar transmittance losses due to dirt and dust (which can be washed off) are generally from three to five percent and are occasionally higher.

Based on the results of the NBS laboratory and field studies, two draft standards for evaluating cover plate materials for flat plate solar collectors were prepared and submitted to ASTM Subcommittee E44.04 on Materials Performance. The titles of the two draft standards are: 1) Standard Practice for Exposure of Cover Materials for Solar Collectors to Natural Weathering Under Conditions Simulating Stagnation Mode; and 2) Standard Practice for Evaluation of Cover Materials for Flat Plate Solar Collectors. The draft standards, with some modifications, have been adopted by ASTM as standard E881-82 and E765-80, respectively.

2.1.3 Parametric Study of Optical Properties

The NBS subsequently conducted a study [5] in which the aforementioned transmittance data were analyzed to determine the influence of air mass and integration technique on Method A of ASTM Standard E424 [4] for measuring solar energy transmittance. As mentioned above, Method A utilizes an integrating sphere spectrophotometer for measuring the transmittance of solar collector cover plates (and also the reflectance of absorber materials). The spectral data so obtained are integrated by arbitrarily selecting one of two methods of integration--weighted ordinates or selected ordinates. One of the objectives of the NBS study was to determine whether the two methods of integration yield the same optical property values for typical collector materials at an air mass equal to 2.0. The second objective was to determine whether a variation in the solar energy spectral distribution (air mass) affects the transmittance and reflectance values of typical materials. The third objective was to develop recommendations for the revision of E424, Method A, if necessary. Three air mass values were used in the calculations: 2.0 (which is specified in Method A of E424), 1.5 and 1.0.

The available transmittance data had been obtained during the field and laboratory study [3] of cover plates described above. The transmittance data used in this study were obtained from: 1) control specimens (no natural or artificial weathering) and 2) specimens exposed to oven aging at 150°C (302°F). The spectral transmittance curves for the control specimens are presented in figures 2.1 and 2.2. Similar spectral transmittance curves are available for the specimens exposed to heat aging.

In the weighted ordinate method of integration, the spectral distribution of sunlight (i.e., solar spectrum) is divided into a finite number of equal intervals, e.g., 50 nm. The relative energy for each interval is calculated by dividing the energy calculated for that interval by the total energy under the solar spectrum curve. The wavelength at the midpoint of the interval is then used as the wavelength for the weighted ordinate. The total solar energy

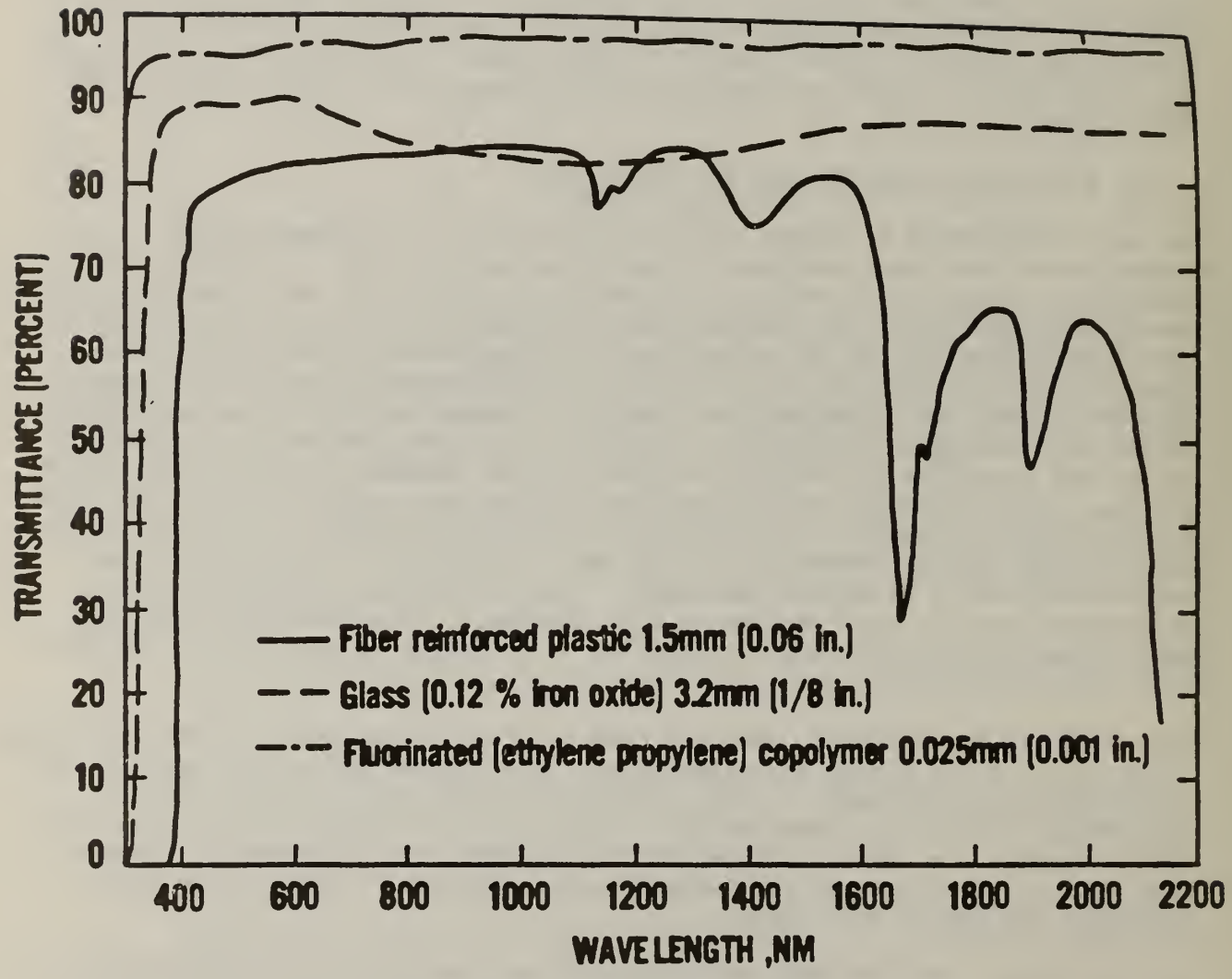

Figure 2.1 Spectral Transmittance of Three Cover Plate Materials, Fiber Reinforced Plastic, Fluorinated (ethylene propylene) Copolymer and Glass (from reference [5])

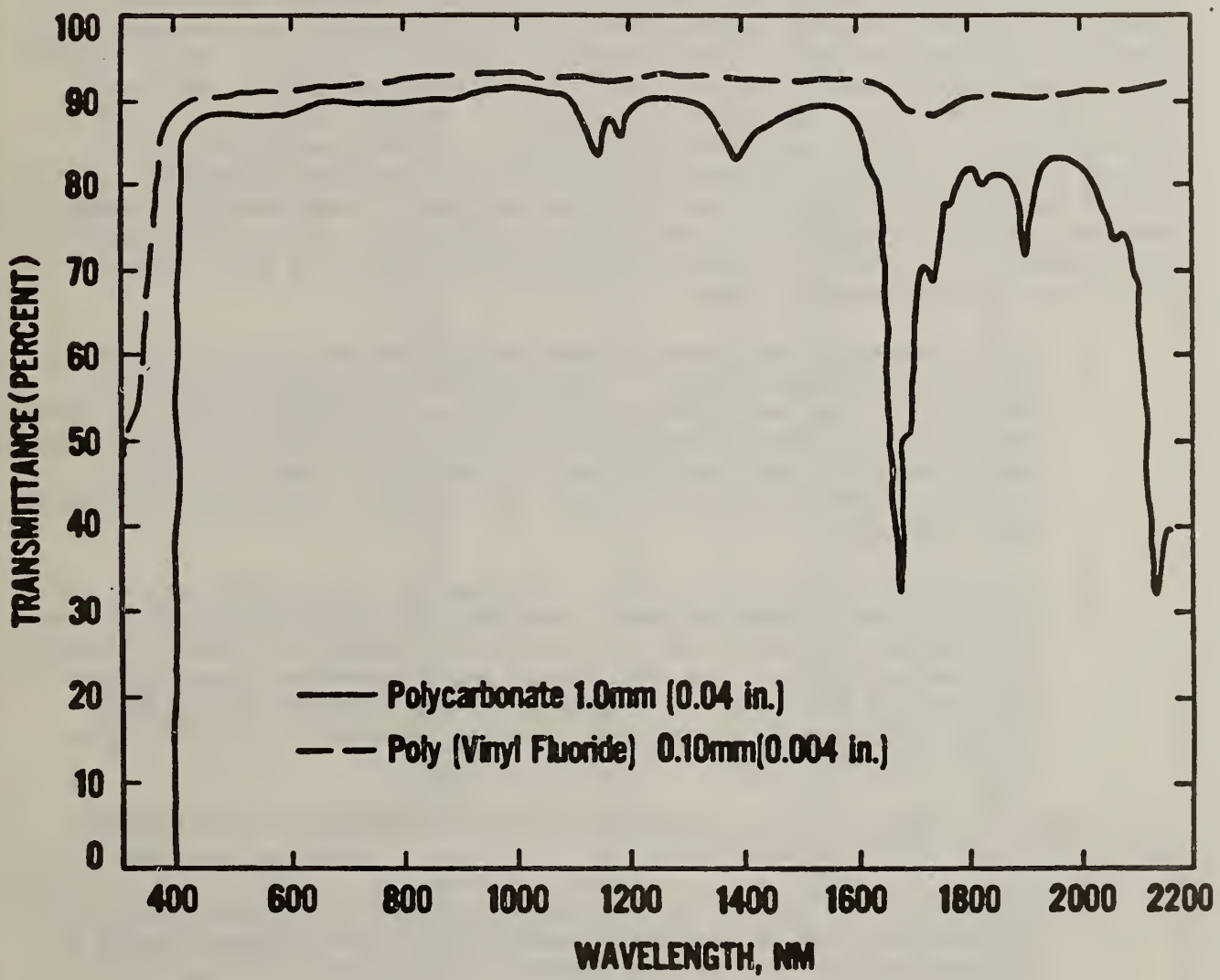

Figure 2.2 Spectral Transmittance of Two Cover Plate Materials, Polycarbonate and Poly(vinyl fluoride) (from reference [5])

transmittance for a given material is then determined by summing the products of transmittances at specific wavelengths times the fraction of the total energy in the interval times the transmittances at a specific wavelength. The 36 specific wavelengths (at 50 nm intervals) and their corresponding relative energy values were taken directly from table A.1 of Standard E424 and used in computing the total solar energy transmittances for each material specimen at air mass 2.0.

In the selected ordinate method, the solar spectrum is divided into a finite number of intervals with equal increments of solar energy. The wavelength at the midpoint of each interval is the selected ordinate. The spectral transmittance of a given cover plate material is thus weighted equally in each interval and no further weighting is necessary. The total solar energy transmittance is determined by summing the materials transmittance value (as obtained from the spectral transmittance curve) for each of the selected ordinates and dividing the sum by the number of selected ordinates. Standard E424 uses 20 selected ordinates for air mass 2.0. These 20 ordinates were used in fulfilling the first objective of the study. In the comparative study of the effects of air mass variation, 50 selected ordinates were used for air masses 1.0 and 1.5 based on data obtained from other studies. Transmittance values that were obtained for the weathered and the control specimens using both methods of integration are presented in table 2.2. Qualitative results are summarized in table 2.3. Upon evaluating the results of the parametric study, two conclusions were drawn:

1. The selected and weighted ordinate methods do not produce identical solar transmittance values. Transmittance can vary by up to one percent for some materials depending on which method is used in the calculations. The differences occur with cover plate materials which have absorption bands in the near infrared (NIR) region of their spectra. Where differences occur, the weighted ordinate method gives higher results.

2. Air mass values used in calculations have a considerable effect on the transmittance values obtained. Changing the air mass values used in calculating transmittances can lead to differences of 3 to 4 percent for some materials. The differences may increase as degradation of the material increases. The materials most affected by air mass change are those which transmit the least energy in the ultraviolet region (below 390 nm).

As a result of this analytical investigation two recommendations were made with respect to ASTM Standard E424-71:

1. Because the two methods of integration do not yield identical results for material having absorption bands in the near infrared region of the spectra, Method A should specify only one method of calculating transmittance.

Table 2.2 Tabulation of Transmittance Data for Cover Plate Materials (from reference [5])

Material	Exposure Test	Time of Exposure Hours	Weighted Ordinate at Air Mass 2.0	Selected Ordinate at Air Mass 2.0	Selected Ordinate at Air Mass 1.5	Selected Ordinate at Air Mass 1.0
Glass	Control	0	87.4	87.6	87.4	87.1
Fluorinated (ethylene propylene) copolymer	Control	0	96.4	96.5	96.3	96.4
Poly(vinyl fluoride)	Control	0	92.2	92.4	91.7	91.3
Polycarbonate	Control	0	88.2	87.3	84.5	82.7
Fiber reinforced plastic	Control	0	78.2	76.8	76.7	72.8
Glass	Heat Age 150°C	10	87.1	87.1	87.2	86.7
		100	87.4	87.6	87.3	87.0
		225	87.1	87.2	87.1	86.5
		500	87.1	87.3	86.9	86.6
		750	87.2	87.3	87.1	86.8
		1000	86.9	86.1	86.9	86.5
		1250	86.6	86.7	86.6	86.1
		1500	87.5	87.6	87.3	86.0
		1825	87.3	87.4	87.2	86.9
Fluorinated (ethylene propylene) copolymer	Heat Age 150°C	10	95.4	95.3	95.3	95.2
		100	95.4	95.4	95.3	95.4
		225	95.5	95.5	95.4	95.3
		500	95.6	95.6	95.6	95.4
		750	95.3	95.4	95.3	95.3
		1000	95.4	95.5	95.5	95.5
		1250	95.6	95.5	95.3	95.4
		1500	95.6	95.5	95.3	95.4
		1825	95.2	95.2	95.0	95.1
Poly(vinyl fluoride)	Heat Age 150°C	10	91.2	91.1	90.5	90.2
		100	87.5	87.3	85.4	84.2
		225	88.3	88.0	86.6	85.4
		500	86.7	86.4	84.6	83.1
		750	82.3	81.8	79.4	77.3
		1000	80.4	80.0	77.2	75.0
		1250	74.9	74.5	71.2	68.7
		1500	71.6	71.2	67.7	65.1
		1825	72.1	71.7	68.2	65.8
Polycarbonate	Heat Age 150°C	10	86.9	86.1	83.4	81.5
		225	87.1	86.2	83.4	81.7
		500	86.7	85.6	82.9	81.0
		750	86.3	85.4	82.6	80.8
		1000	86.5	85.6	82.7	80.9
		1250	86.1	85.3	82.4	80.5
		1500	86.3	85.3	82.5	80.6
		1825	85.6	84.8	81.9	80.0
Fiber reinforced plastic	Heat Age 150°C	10	70.9	69.7	67.2	65.2
		100	60.6	59.8	56.6	54.4
		225	54.3	53.5	50.2	48.0
		500	52.4	51.7	48.4	46.3
		750	43.9	43.3	40.4	38.4
		1000	40.0	39.5	36.7	35.0
		1250	40.0	39.4	36.7	34.9
		1500	44.8	44.2	41.0	39.1
		1825	39.7	39.1	36.5	34.7

Table 2.3 Qualitative Results of Parametric Study of Cover Plate Materials (from reference [5])

Cover Plate Materials	Effect on Transmittance of: Integration Methods	Effect on Transmittance of: Air Mass
Fiber-reinforced plastic	Selected ordinate method yielded lower values of transmittance*.	Transmittance decreased with decreasing air mass*.
Poly-carbonate	Selected ordinate method yielded lower values of transmittance*.	Transmittance decreased with decreasing air mass*.
Poly(vinyl fluoride)	1) No difference for unweathered 2) Selected ordinate method yielded lower values for weathered specimens.	Transmittance decreased with decreasing air mass*.
Fluorinated Ethylene propylene	No difference*	No difference for weathered specimens.**
Glass	No difference*	No difference for weathered specimens.**

* Applies to both weathered and unweathered specimens.

** For unweathered cover plate materials, the transmittance decreased as the air mass decreased.

2. Since most collectors in service are exposed to more hours of radi[illegible] at air mass 1.5 than at air mass 2.0, the solar energy distributi[illegible] used in Method A should be changed to air mass 1.5.

These proposed revisions are still under consideration by ASTM Committee E44 on Solar Energy Conversion.

2.1.4 Weathering Performance of Cover Materials

In order to obtain somewhat longer term natural weathering data on cover plate materials, the NBS extended the exposure periods of the minicollectors mentioned in section 2.1.2 for up to 24 months [6]. In addition to providing a measure of the relative durabilities of the ten cover plate materials (see table 2.1), the natural weathering data served as a basis of comparison for data obtained from the artificial weathering with a xenon arc light. The comparative analysis of the two methods of weathering was done with a view toward the development of uniform methods of measuring long-term performance of cover plate materials.

To facilitate the comparison of the effects of natural weathering with those of artificial weathering, the energy deposited on materials during xenon arc exposure was calculated. The hours of xenon arc exposure within the artificial weathering device and the corresponding calculated energy values are shown in table 2.4. The exposure schedule and the solar radiation accumulated during exposure at the three natural weathering sites are summarized in table 2.5. The solar energy transmittance of the material specimens was determined using method A of ASTM E424-71. For the natural weathering exposure tests, the transmittance was obtained for both single and double cover minicollectors. The transmittance of the field-weathered specimens was measured both before and after a cleaning procedure to determine the effect of dirt accumulation on the optical properties. The results of the field study were presented in tabular and graphical forms, showing the percent of solar energy transmittance as a function of exposure time. A typical set of test curves is shown in figure 2.3. The solar energy transmittances of the inner and outer covers of the minicollectors were graphically compared with that from the specimens exposed to the xenon arc light. One such comparative curve is shown in figure 2.4.

In addition to measuring the effect of weathering on optical properties, the effect on the tensile properties of three plastic films was measured using ASTM D882, Tests for Tensile Properties of Thin Plastic Sheeting. The tensile properties which were reported were: 1) breaking factor, 2) tensile strength, 3) elongation at break, 4) yield strength, and 5) elongation at yield. It was concluded that the evaluation process for cover plate materials should include measurement of mechanical as well as optical properties. Further, it was concluded that tensile strength and elongation at break are useful parameters for observing changes in the mechanical properties of plastic film materials caused by weathering.

The results of the additional two years of natural weathering strengthened the previous conclusion that minicollectors adequately simulated the conditions

Table 2.4 Calculated Values for Energy Deposited on Cover Materials During Artificial Weathering with Xenon Arc Light (from reference [6])

Hours of Exposure	Energy Deposited (Calculated) GJ/m^2
250	1.030
499	2.055
758	3.122
1000	4.118
2012	8.286
3000	12.355
4010	16.515

Table 2.5 Natural Weathering Exposure Data for Minicollectors (from reference [6])

		Solar Radiation (GJ/m^2)		
Months Exposure	Dates	Gaithersburg, Maryland	Miami, Florida	New River, Arizona
3	9/1/77-12/1/77	1.176	1.455	2.012
	12/20/77-3/20/78			1.422
6	9/1/77-3/1/78	2.251	2.864	3.487
	3/24/78-9/24/78			5.123
12	9/1/77-9/1/78	5.477	6.344	8.006
	3/1/78-3/1/79	5.429		
	4/1/78-4/1/79		6.523	
	7/29/78-7/29/79			8.259
18	9/1/77-3/1/79	7.680	9.333	11.642
24	9/1/77-9/1/79	10.720	13.125	16.370
36	9/1/77-9/1/80	16.402	19.398	24.414
	4/1/78-4/1/81		19.859	
	4/17/78-4/17/81	16.693		
48	9/1/77-9/1/81	22.076	26.333	33.038

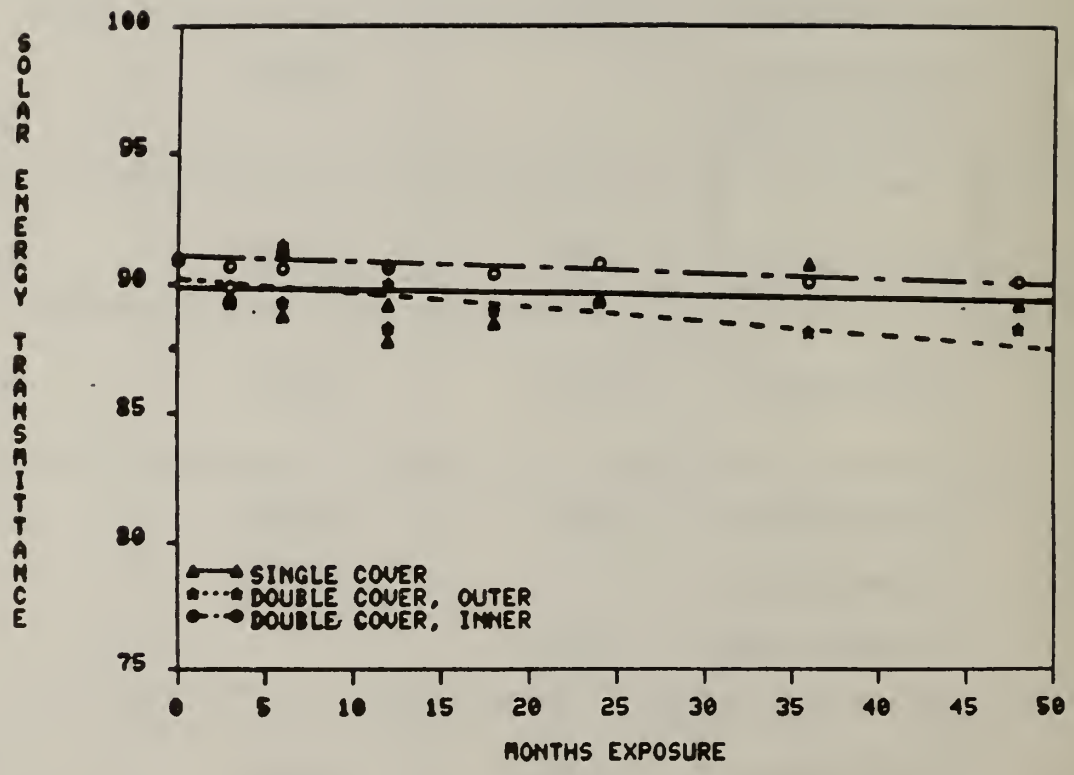

Figure 2.3 Solar Energy Transmittance of Poly(methyl methacrylate) After Weathering on Minicollectors in New River, Arizona

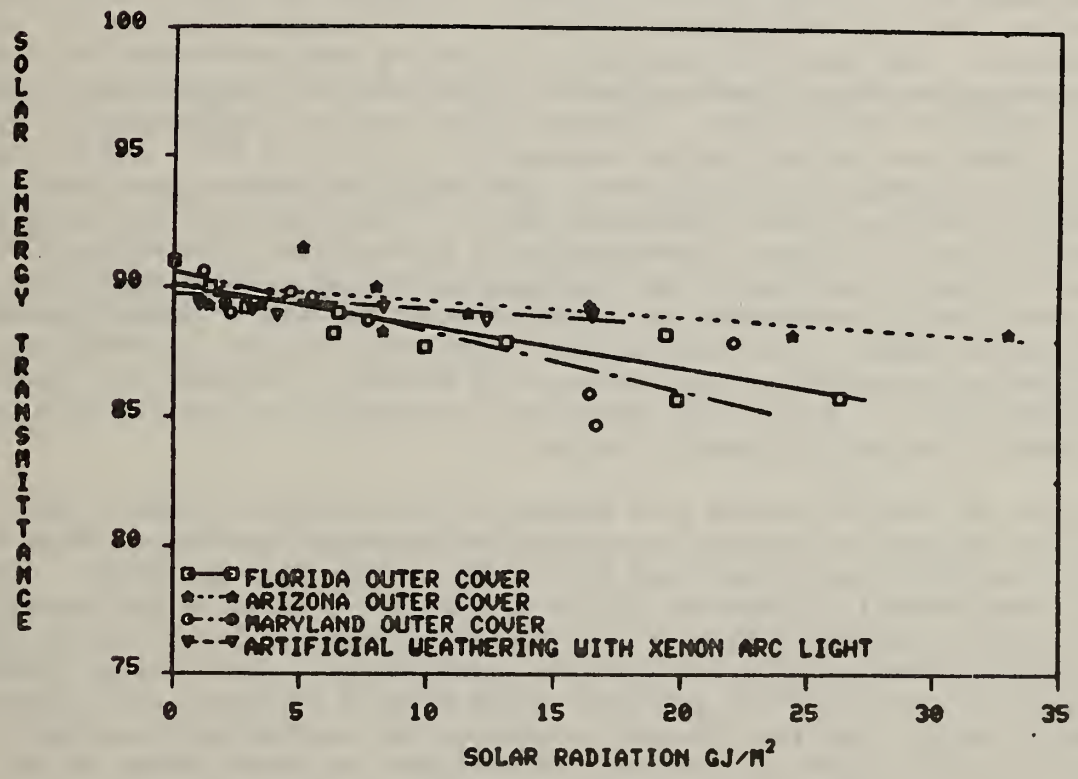

Figure 2.4 Comparison of Solar Transmittance of Poly(methyl methacrylate) After Weathering on a Minicollector as an Outer Cover and In an Artificial Weathering Device with Xenon Arc Light (from reference [6])

encountered by cover plates on some flat plate collectors. Furthermore, the mechanical and optical property data associated with the artificial weathering with xenon arc light strengthened the case for this laboratory method of distinguishing materials sensitive to natural sunlight.

2.1.5 Hail Impact Testing

As implied in section 2.1.1, the physical deterioration of cover plates can substantially degrade the efficiency of the solar collector subsystem. In order to effectively perform their intended optical and thermal functions, cover plates must be able to resist or be protected from forces which can cause rupture, cracking, extensive crazing or surface distortion. One of the main environmental forces that can cause failure of a cover plate is hail impact. Although some solar building construction standards required hail resistance testing, there was no standard testing procedure for glazing materials. The NBS conducted a laboratory study [7] aimed at developing such a standard test method. Thus, the primary purpose of the research was to evaluate and refine previously-used procedures toward the development of a standard test method for simulating hail impact on solar collector covers.

In this study, ice balls of various diameters and velocities were propelled by a compressed air launcher against targets consisting of typical cover plate materials. The ice ball parameters used in the laboratory testing are given in table 2.6. The materials selected for testing were considered to be representative of the three classes of cover plates normally encountered, namely, rigid, semirigid and flexible. Tempered glass was used to represent a rigid cover. Fiber reinforced plastic represented a semirigid cover and polymeric thin film represent a flexible cover. The principal factors considered in evaluating the impact test procedures were: 1) velocities of ice balls used to simulate hail, 2) impact loads caused by falling hail versus propelled ice balls, 3) impact locations on the collector cover, 4) support conditions for the covers, and 5) assessment of collector cover failure or damage caused by impacting ice balls. Some experimental effort was devoted to developing: 1) a procedure for preparing the ice balls, 2) a method of aligning the launcher on the target area, 3) a method of measuring the velocity of the ice ball, and 4) a means of measuring kinetic forces.

The glass and plastic sheets were mounted in cover support frames. One type of frame was designed to support both rigid and semirigid specimens, while a slightly modified design was used for flexible, thin film materials. The frames were sized to accomodate 1.93 m by 0.86 m (76 in by 34 in) sheets; this frame size was representative of flat plate collectors. To provide a basis for comparing results obtained with the standardized support frame, several specimens of tempered glass were mounted in a solar collector and subjected to ice-ball impact. The test procedure involved the successive propulsion of ice balls of various diameters and velocities against selected points on the mounted plate material. Seventeen points of impact were marked on the exposed face of the sheet. For a given specimen, an initial iceball size and corresponding velocity were selected which were not expected to cause failure. If there was no visible damage for the initial set of parameters, the next larger size ice ball and corresponding velocity were used for impact. On the other

Table 2.6 Ice Ball Parameters for Laboratory Testing (from reference [7])

Diameter (in.)	Mass (lbm)	Velocity[1/] Terminal (ft/s)	Velocity[1/] Resultant[2/] (ft/s)	Kinetic Energy[3/] Terminal (ft-lbf)	Kinetic Energy[3/] Resultant (ft-lbf)	Momentum[3/] Terminal (lbf-s)	Momentum[3/] Resultant (lbf-s)
3/4	0.0073	62	91	0.44	0.94	0.014	0.021
1	0.0173	73	98	1.43	2.58	0.039	0.053
1-1/4	0.0337	82	105	3.52	5.77	0.086	0.110
1-1/2	0.0584	90	112	7.35	11.38	0.163	0.203
1-3/4	0.0928	97	117	13.56	19.73	0.280	0.337
2	0.1384	105	124	23.69	33.04	0.451	0.533
2-1/4	0.1971	111	129	37.71	50.93	0.679	0.790
2-1/2	0.2705	117	134	57.50	75.42	0.983	1.126

1/ For laboratory testing, ice balls were propelled in a direction perpendicular to the plane of impact.

2/ Resultant velocity is the vector sum of the terminal velocity and a 66 ft/s horizontal wind component.

3/ Corresponding to the terminal and resultant velocity.

Table 2.7 Summary of Ice Ball Impact Test Results (from reference [7])

Material	Specimen Number	Appendix Table	Ice Ball Causing Failure: Diameter (in.)	Ice Ball Causing Failure: Velocity[1] (ft/s)	Ice Ball Causing Failure: Kinetic Energy (ft-lbf)	Impact Location[2] and Type of Failure	Prior Impact Tests	Edge Loading[3] (lbm/ft)
Tempered glass. Exposed side textured, other side smooth. (1/8 in. thick)	#1	A-3	1-3/4	119 R	20	#1 Fracture	1 R, 1-1/2 R	-
	#2	A-5	1-3/4	115 R	19	#2 Fracture	1-3/4 T	-
	#3	A-6	1-3/4	122 R	20	#2 Fracture	None	-
	#4	A-7	1-3/4	101 R	14	#2 Fracture	None	-
	#5	A-8	1-1/2	95 T	7.5	#2 Fracture	None	-
	#6	A-11	1-3/4	98 T	13	#2 Fracture	1-1/2 T,1-1/2 R	-
Tempered glass. Both sides smooth. (1/8 in. thick)	#1	A-13	2	129 R	36	#14 Fracture	2 T	-
	#2	A-17	2-1/4	131 R	49	#5 Fracture	2 R, 2-1/4 T	-
	#3	A-18	2-1/4	127 R	47	#3 Fracture	None	-
	#4	A-20	2-1/4	114 T	38	#9 Fracture	2 T	-
	#5	A-21	2	130 R	34	#4 Fracture	None	-
	#6	A-22	2	128 R	33	#1 Fracture	None	-
Tempered glass. Both sides smooth. (1/8 in. thick) Mounted in Solar Collector	A	A-38	2-1/2	135 R	78	#1 Fracture	1-3/4 R, 2 R	-
	F	A-39	1-3/4	119 R	20	#1 Fracture	None	-
	B	A-40	2-1/2	136 R	80	#10 Fracture	2 R	-
	E	A-41	2-1/2	136 R	79	#10 Fracture	2 R	-
	C	A-42	2-1/2	137 R	80	#14 Fracture	1-3/4 R, 2 R	-
	D	A-43	1-3/4	115 R	19	#14 Fracture	None	-
Reinforced plastic. Polyester resin with glass fiber mat. (0.040 in. thick)	#1	A-23	1-1/2	90 T	5.2-9.2	#1 to #11, Leak	None	-
	#1	A-23	1-1/4	82 T	3.1-5.3	#12 to #17, Leak	None	-
	#2	A-24	3/4	62 T	0.38-0.53	#1 to #17, Cracks	None	-
	#3	A-25	1-1/4	82 T	3.0-3.9	#1, #3 to 6, #8 to 11, #13 to 16, Leak	None	-
	#4	A-26	1-1/4	105 R	4.4-5.9	#2 to #12, #14, #15, Leak, #16, #17, Break	None	-
	#5	A-27	1-1/2	98 R	8.3-11.7	#1 to #17, Break	None	-
	#6	A-28	1-1/4	82 T	2.9-3.7	#3 to #9, Leak	None	-
	#7	A-29	1-3/4	97 T	12-18	#1 to #9, Break	None	-
Poly(vinyl fluoride) film. (0.004 in. thick)	#1	A-30	3/4	62 T	0.36-0.49	#1 to #17, Slight damage, small indentations	None	3.3 side 4.2 end
	#1	A-31	3/4	91 R	0.76-1.01	#1 to #17, Slight damage, small indentations	3/4 T	
	#2	A-32	1	73 T	1.1-1.6	#1 to #17, Slight damage, indentations	None	3.6 side 5.3 end
	#2	A-33	1	98 R	2.2-2.7	#1 to #17, Slight damage, indentations	1 T	
	#3	A-34	1-1/4	82 T	2.9-4.0	#1 to #4, #7 to #9, Indentations	None	5.2 side 5.5 end
			1-1/4	105 R	5.0-5.7	#5, #6, #10 to #12, #16, #17, Indentations		
	#3	A-34	1-1/2	90 T	5.8-6.6	#13, #14, Indentations	None	
			1-1/2	112 R	9.3	#15 Indentation		
	#4	A-35	2	105 T	19-24	#2, #6 to #9, Break	None	5.2 side 5.5 end
					18-20	#1, #3 to #5, Indentations		
	#5	A-36	1-3/4	117 R	16	#1, #9, Indentations	None	5.2 side
					17-18	#2, #3, #6 to #8, Break		5.5 end
	#6	A-37	1-3/4	97 T	11-15	#1 to #9, #11 to #13, Indentations		5.2 side 5.5 end
					12-15	#10, #14 to #17, Break	None	

1/ R = resultant velocity, T = terminal velocity.
2/ Locations are denoted by a number as shown in figure 20 in reference [7].
3/ Edge loading of plastic film during mounting in the cover support frame.

hand, a damaged specimen was immediately discarded. This procedure was repeated with ice balls of increasing size until failure occurred.

A summary of results from tests on four solar collector cover materials is presented in table 2.7. The table reports: 1) diameter, velocity, and kinetic energy of the ice ball causing failure; 2) location of impact and type of failure; 3) prior impact tests on the tempered glass specimens; and 4) the amount of tensile edge loading of plastic film specimens during mounting in the cover support frame.

The laboratory experiments confirmed the feasibility of simulating the impact of hailstones by propelling ice balls from a compressed air launcher with interchangeable plastic barrels. It was concluded that the ice balls should impact a specimen along a path perpendicular to the specimen surface at a resultant velocity which is the vector sum of the hailstone terminal velocity and a horizontal wind velocity component equal to 72 km/h (45 mi/h). The investigators emphasized the need for a system for accurate velocity measurement of ice balls in flight. Information from this study was used to prepare a draft standard for consideration by ASTM Subcommittee E44.04 on Materials Performance. Subsequently, the ASTM Task Group on Cover Plates, E44.04.02, prepared a draft standard practice which was adopted as E822-81, "Practice for Determining Resistance of Solar Collector Covers to Hail by Impact with Propelled Ice Balls."

2.2 ABSORBER PLATES

2.2.1 Introduction

The purpose of absorber plates in solar energy systems is to convert solar energy into useful thermal energy. For optimum efficiency, the solar absorber should possess maximum possible absorptance (α) in the solar spectrum while exhibiting minimum infrared emittance (ε). Generally, high absorptance in the ultraviolet, visible, and near infrared portions of the spectrum is obtained by a black surface coating. Low infrared emittance is obtained by using a highly reflective metal substrate beneath the coating. A coating material which captures solar energy in the high intensity visible and near infrared spectral regions while maintaining poor infrared radiating properties is called a selective coating.

Much of the research conducted since 1974 on absorber components has been directed at: 1) understanding and modeling the microstructure of selective coating candidate materials, 2) improving optical properties (i.e., absorptance and emittance) of absorbers by studying the performance of various combinations of coatings and substrate material, and 3) establishing standard test procedures for evaluating the performance of absorbers under conditions of high temperature and humidity (for both short- and long-term operation). Several national laboratories have engaged in studies to evaluate the performance of specific absorber coating/substrate combinations under simulated normal operating and stagnation conditions. Black chrome has been extensively studied as an absorptive coating because of its desirable properties.

2.2.2 Laboratory and Field Experimentation

The lack of short-term and long-term performance data on absorber materials in operating collectors and the lack of standardized test methods for the prediction of long-term performance have hampered efforts to derive solutions to some documented collector problems. Of particular concern are the effects of elevated temperature due to stagnation, thermal cycling, ultraviolet radiation, and moisture. The National Bureau of Standards conducted studies [8,9] on the performance and durability of several absorber materials to aid in the development of standardized, accelerated-aging test methods. It was intended that the test methods be used to predict the long-term performance of absorptive coatings for solar collectors.

Laboratory and field aging tests were conducted on twelve absorber materials used primarily in building heating, cooling, and domestic hot water applications. Table 2.8 tabulates all of the absorber materials and the substrates on which they were applied. Absorptance and infrared emittance were determined using ASTM E424-71, Method A and ASTM E408-71 respectively. The optical property measurements were made before and after the aging tests to determine the deleterious effects of the aging processes. In addition to optical measurements, the specimens were visually inspected before and after aging for signs of loss of adhesion properties and changes in visual appearance. The aging tests included accelerated laboratory exposure and outdoor exposure in simulated solar collectors. The two-pronged test program has as one of its objectives to determine whether or not the data obtained by the two methods of tests lead to the same level of degradation of absorptive coatings.

In the accelerated aging tests, the materials were exposed to elevated temperatures, thermal cycling, ultraviolet radiation (UV) and moisture. Test specimens were exposed in an oven to elevated temperatures of 150, 200 and 250°C and removed at various time increments for measurement of properties. The maximum time of exposure was 14 weeks for the 150°C specimens, while the remaining specimens were exposed for a maximum of 12 weeks. In the thermal cycling tests, specimens were exposed to 84 cycles with each cycle consisting of 7.5 hours at 200°C followed by 15.5 hours at -10°C. Ultraviolet radiation exposure was simulated through the use of ASTM G26-77, in which a xenon arc accelerated weathering machine of measured solar intensity is specified. Specimens were exposed for time increments ranging from 125 to 2100 hours before measurement was performed. Test specimens were exposed in a humidity cabinet at 92°C and 97 percent relative humidity and removed at time increments of 1,2, 6 and 21 weeks for property measurement. In addition to the single aging tests, a cumulative exposure test was performed. In the cumulative exposure test, specimens were sequentially exposed to elevated temperature, thermal cycling, UV radiation, and moisture. Optical properties were measured after each exposure condition.

Absorptance and infrared emittance values were tabulated for the above-mentioned accelerated exposure tests, thereby facilitating comparison with optical property measurements obtained for unaged specimens. The average initial absorptance and emittance values obtained from ten unaged specimens of each absorber material are shown in table 2.9. As an example of the accelerated aging test

Table 2.8 Absorber Materials and Substrates Used in the Study (from reference [8])

Code Number[1/]	Absorber Material	Substrate
A8	Nickel coated foil	Aluminum, alodine
B6	Anodized aluminum	Aluminum, 6061, anodized
B7	Anodized aluminum	Aluminum, 1100, anodized
C5	Copper oxide	Copper
D2	Black chrome	Steel, cold-rolled[2/]
D4	Black chrome	Copper, DHP 122[3/]
G9	Polyvinylidene fluoride	20 gage, G90, hot dipped galvanized steel
H1	Polyvinylidene fluoride	Steel, bonderized
H8	Polyvinylidene fluoride	Aluminum, alodine
I1	Urethane	Steel, bonderized
I4	Urethane	Copper, DHP 122
I8	Urethane	Aluminum, alodine
J1	Alkyd	Steel, bonderized
J4	Alkyd	Copper, DHP 122
J8	Alkyd	Aluminum, alodine
K1	Epoxide	Steel, bonderized
K4	Epoxide	Copper, DHP 122
K8	Epoxide	Aluminum, alodine
L1	Silicone	Steel, bonderized
L4	Silicone	Copper, DHP 122
L8	Silicone	Aluminum, alodine
M1	Modified polyester	Steel, bonderized
M4	Modified polyester	Copper, DHP 122
M8	Modified polyester	Aluminum, alodine
N2	Porcelain enamel	Steel, cold-rolled
N8	Porcelain enamel	Aluminum, alodine

1/ The letter refers to the absorber material and the number to the substrate.
2/ Includes 0.5 mil nickel flash.
3/ Includes 0.1 - 0.2 mil nickel flash.

Table 2.9 Absorptance and Infrared Emittance of Absorbers Prior to Exposure (from reference [8])

Code Number	Absorber Material	Absorptance (α)	Emittance (ε)
A8	Nickel foil	0.98	0.09
B6	Anodized aluminum	0.92	0.81
B7	Anodized aluminum	0.90	0.82
C5	Copper oxide	0.95	0.74[1/]
D2	Black chrome	0.95	0.07
D4	Black chrome	0.96	0.06
G9	Polyvinylidene fluoride	0.95	0.85
H1	Polyvinylidene flouride	0.96	0.85
H8	Polyvinylidene fluoride	0.96	0.88
I1	Urethane	0.97	0.92
I4	Urethane	0.97	0.92
I8	Urethane	0.97	0.92
J1	Alkyd	0.97	0.92
J4	Alkyd	0.97	0.91
J8	Alkyd	0.97	0.92
K1	Epoxide	0.97	0.91
K4	Epoxide	0.97	0.91
K8	Epoxide	0.97	0.91
L1	Silicone	0.97	0.88
L4	Silicone	0.97	0.87
L8	Silicone	0.97	0.88
M1	Modified polyester	0.93	0.86
M4	Modified polyester	0.89	0.84
M8	Modified polyester	0.90	0.81
N2	Porcelain	0.93	0.85
N8	Porcelain	0.93	0.84

1/ Emittance for 10 specimens of this sample varied from 0.65 to 0.80.

Table 2.10 Results of Thermal Cycling [1/] (from reference [8])

Code Number	Absorber Material	NUMBER OF CYCLES											
		Absorptance (α)						Emittance (ε)					
		0	7	15	28	42	84	0	7	15	28	42	84
A8	Nickel Foil	.98	.98	.98	.98	.99	.98	.09	.09	.10	.09	.09	.11
B6	Anodized Aluminum	.92	.91	.91	.92	.91	.92	.81	.81	.81	.81	.81	.82
B7	Anodized Aluminum	.90	.90	.89	.90	.90	.90	.82	.81	.81	.81	.82	.82
C5	Copper Oxide	.95	.90	.88	.85	.82	.86	.74	.70	.68	.64	.54	.66
D2	Black Chrome	.95	.95	.96	.96	.94	.94	.07	.09	.06	.06	.06	.07
D4	Black Chrome	.96	.94	.95	.94	.94	.92	.06	.06	.06	.06	.06	.05
G9	Polyvinylidene Fluoride	.95	.95	.95	.95	.95	.95	.85	.84	.84	.84	.86	.91
H1	Polyvinylidene Fluoride	.96	.95	.96	.96	.96	.96	.85	.83	.84	.80	.82	.81
H8	Polyvinylidene Fluoride	.96	.95	.96	.96	.96	.96	.88	.89	.88	.89	.87	.85
I1	Urethane	.97	.97	.97	.98	.97	+	.92	.92	.92	.93	.94	+
I4	Urethane	.97	.97	.97	.98	.97	.97	.92	.92	.93	.93	.94	.94
I8	Urethane	.97	.97	.97	.8	.98	.97	.92	.93	.93	.93	.94	.94
J1	Alkyd	.97	.97	.97	.93	.97	+	.92	.91	.91	.90	.91	+
J4	Alkyd	.97	.97	.97	+	+	+	.91	.91	.91	+	+	+
J8	Alkyd	.97	.97	.97	.97	.97	.97	.92	.91	.99	.91	.92	.91
K1	Epoxide	.97	.97	.97	.97	+	+	.91	.90	.91	.90	+	+
K4	Epoxide	.97	.97	.97	+	+	+	.91	.90	.91	+	+	+
K8	Epoxide	.97	.97	.97	.97	.97	.97	.91	.90	.91	.91	.91	.91
L1	Silicone	.97	.97	.97	.97	.97	+	.88	.87	.87	.88	.88	+
L4	Silicone	.97	.97	.97	.97	.97	.97	.87	.87	.88	.88	.88	.89
L8	Silicone	.97	.97	.97	.97	.97	.97	.88	.87	.88	.88	.88	.89
M1	Mod. Polyester	.93	.91	.90	.92	+	+	.86	.87	.87	.87	+	+
M4	Mod. Polyester	.89	+	+	+	+	+	.84	+	+	+	+	+
M8	Mod. Polyester	.90	.88	.89	.88	+	+	.81	.86	.87	.86	+	+
N2	Porcelain	.93	.93	.94	.94	.94	.94	.85	.85	.85	.85	.85	.85
N8	Poreclain	.93	.92	.94	.93	.93	.93	.84	.83	.84	.84	.84	.84

1/ + designates removal of absorber material during exposure.

results, table 2.10 presents the results of the thermal cycling exposures. The draft standard that was developed as a result of the laboratory aging tests was subsequently adopted by ASTM as standard E744-80 [10].

In the field aging study, specimens were exposed in test boxes to simulate stagnation conditions at three sites: 1) Gaithersburg, Maryland; 2) Phoenix, Arizona; and 3) Miami, Florida, for periods ranging from twenty to twenty-four months. During these exposure intervals specimens were retrieved for laboratory evaluation of their optical properties and then returned to the field for continuing exposure. The solar absorptance and infrared emittance were measured after approximately 4-month exposure intervals. The field exposure study extended over a period of about three calendar years, beginning in the summer of 1978 and terminating in the fall of 1981.

Triplicate specimens of each coating/substrate combination were exposed in accordance with a method that, with some modifications, was adopted by ASTM as E781-81 [11]. The exposure boxes simulated solar collectors of single cover plate construction. The orientation (tilt angle) of the exposure boxes was adjusted four times each year to provide increased solar flux. Table 2.11 shows the results of the outdoor exposures at Miami, Florida. The total direct solar irradiation received during the 24 months of exposure was 13.62×10^9 J/m^2.

The comparison of data obtained from the laboratory tests with those from the field exposure tests led to the conclusion that: "Degradation of absorptive coatings, such as change of optical properties or appearance that was observed during outdoor exposures, was also observed in one or more of the accelerated laboratory exposures included in ASTM E744-80. This indicates that the degradation processes induced by stagnation exposure are adequately addressed by the accelerated laboratory exposures." The significance of this conclusion lies in the fact that ASTM E781-81 is now one of the testing options mentioned by E744-80.

2.2.3 Parametric Study of Optical Properties

In the above mentioned parametric study of cover plates (section 2.1.3) [5], NBS also analyzed laboratory test data on the performance of absorber materials. The purpose of the study was to assess the effect of several variables on an ASTM standard method for measuring the reflectance of absorber materials (Method A of ASTM E424). In Method A of E424 [4], an integrating sphere spectrophotometer is used to measure the spectral reflectance of materials. The spectral data so obtained is integrated by arbitrarily selecting one of two methods of integration: weighted ordinates or selected ordinates. One of the objectives of the NBS study was to determine whether the two methods of integration yield identical optical property values for typical absorber materials at air mass equal to 2.0. The second objective was to determine whether the variation in the solar energy spectral distribution (i.e., air mass) affects the reflectance values of typical absorber materials. The third objective was to develop recommendations for the revision of E424, Method A, if necessary.

Table 2.11 Results of Outdoor Exposure at Miami, Florida

Cumulative Time of Exposure (Months) (from reference [9])

Code Number	Absorptive Coating	Absorptance (α) Months 0	4[1]	8[2]	12[3]	16[4]	20[5]	24[6]	Emittance (ε) Months 0	4[1]	8[2]	12[3]	16[4]	20[5]	24[6]
A8	Nickel Foil	0.98	0.99	0.98	0.98	0.98	0.98	0.97	0.09	0.05	0.08	0.08	0.08	0.09	0.10
B6	Anodized Aluminum	0.92	0.92	0.92	0.91	0.91	0.91	0.91	0.81	0.81	0.81	0.81	0.81	0.81	0.81
B7	Anodized Aluminum	0.90	0.90	0.90	0.89	0.89	0.89	0.90	0.82	0.81	0.80	0.81	0.81	0.80	0.81
C5	Copper Oxide	0.95	0.90	0.89	0.88	0.88	0.87	0.86	0.74[7]	0.50	0.53	0.52	0.52	0.53	0.54
D2	Black Chrome	0.95	0.97	0.96	0.95	0.95	0.95	0.94	0.07	0.04	0.07	0.06	0.06	0.07	0.08
D4	Black Chrome	0.96	0.94	0.93	0.92	0.92	0.92	0.91	0.06	0.06	0.06	0.06	0.05	0.06	0.07
G9	Polyvinylidene Fluoride	0.95	0.95	0.95	0.95	0.95	0.95	0.94	0.85	0.85	0.86	0.85	0.85	0.84	0.84
H1	Polyvinylidene Fluoride	0.96	0.95	0.95	0.95	0.95	0.95	0.95	0.85	0.82	0.85	0.83	0.84	0.83	0.83
H8	Polyvinylidene Fluoride	0.96	0.95	0.96	0.95	0.95	0.95	0.95	0.88	0.87	0.88	0.87	0.87	0.86	0.86
I1	Urethane	0.97	0.97	0.97	0.97	0.97	0.97	0.96	0.92	0.92	0.92	0.93	0.93	0.93	0.93
I4	Urethane	0.97	0.97	0.97	0.97	0.97	0.97	0.96	0.92	0.92	0.93	0.93	0.93	0.93	0.93
I8	Urethane	0.97	0.97	0.97	0.97	0.97	0.97	0.96	0.92	0.92	0.92	0.93	0.93	0.93	0.93
J1	Alkyd	0.97	0.98	0.98	0.98	0.98	0.98	0.97	0.92	0.91	0.91	0.91	0.90	0.90	0.90
J4	Alkyd	0.97	0.98	0.98	0.98	0.98	0.98	0.97	0.91	0.91	0.91	0.91	0.91	0.91	0.91
J8	Alkyd	0.97	0.97	0.98	0.98	0.98	0.98	0.97	0.92	0.91	0.91	0.91	0.91	0.91	0.91
K1	Epoxide	0.97	0.98	0.98	0.98	0.98	0.98	0.97	0.91	0.91	0.91	0.90	0.90	0.90	0.90
K4	Epoxide	0.97	0.98	0.98	0.98	0.98	0.98	0.97	0.91	0.91	0.91	0.90	0.90	0.90	0.90
K8	Epoxide	0.97	0.98	0.98	0.98	0.98	0.98	0.97	0.91	0.90	0.91	0.90	0.90	0.90	0.90
L1	Silicone	0.97	0.97	0.97	0.96	0.96	0.96	0.96	0.88	0.87	0.87	0.87	0.87	0.87	0.87
L4	Silicone	0.97	0.97	0.97	0.96	0.96	0.96	0.96	0.87	0.87	0.87	0.87	0.87	0.87	0.87
L8	Silicone	0.97	0.97	0.97	0.97	0.97	0.96	0.96	0.88	0.88	0.85	0.87	0.87	0.88	0.88
M1	Mod. Polyester	0.93	0.94	0.95	0.95	0.94	0.94	0.94	0.86	0.89	0.92	0.91	0.91	0.92	0.92
M4	Mod. Polyester	0.89	0.91	0.93	0.93	0.93	0.93	0.92	0.84	0.85	0.90	0.90	0.90	0.90	0.91
M8	Mod. Polyester	0.90	0.92	0.94	0.94	0.94	0.93	0.93	0.81	0.88	0.92	0.91	0.91	0.93	0.93
N2	Porcelain	0.93	0.94	0.94	0.93	0.94	0.91	0.93	0.85	0.85	0.85	0.85	0.84	0.85	0.85
N8	Porcelain	0.93	0.93	0.92	0.92	0.92	0.93	0.92	0.84	0.84	0.85	0.84	0.84	0.84	0.84

[1] Exposed from 6/78 to 10/78.
[2] Exposed from 11/78 to 5/79 (with a 2 mo. non-exposure period)
[3] Exposed from 7/79 to 11/79.
[4] Exposed from 5/80 to 9/80.
[5] Exposed from 12/80 to 4/80.
[6] Exposed from 5/81 to 9/81.
[7] Emittance for 10 specimens of this sample varied from 0.65 to 0.80.

The available reflectance data had been obtained during the laboratory/field tests on absorber plates described above [5]. The reflectance data were obtained from: 1) control specimens (no natural or artificial weathering), 2) specimens exposed to oven aging, and 3) specimens exposed to humidity at elevated temperatures. Four absorber materials, two selective and two nonselective, were chosen: 1) black chrome on steel (selective), 2) black chrome on copper (selective), 3) urethane paint on steel (nonselective), and 4) porcelain enamel on steel (nonselective). The spectral reflectance curves for the control specimens are presented in figures 2.5 and 2.6. Similar reflectance curves are available for specimens exposed to aging. For calculating the reflectance values, three air masses were used: 2.0 (which is specified in Method A of E424), 1.5 and 1.0. The reader is referred to section 2.1.3 in which brief descriptions of the weighted ordinate and the selected ordinate methods of integration, as applied to transmittance calculations, are presented. The methods were used in an identical manner to obtain reflectance values.

Reflectance values that were obtained for the weathered and the control specimens, using both methods of integration, are presented in table 2.12. Qualitative results are summarized in table 2.13. Upon evaluating the results of the parametric study, three conclusions were drawn:

1. At air mass 2 both the weighted and selected ordinate calculation methods yield comparable reflectance values.

2. A change in air mass has no observable effect on the reflectance value calculated for nonselective absorbers.

3. Air mass values used in calculating reflectance can lead to discrepancies for selective absorbers. Black chrome reflectance values increased by up to 1.2 percent when the air mass changed from 2.0 to 1.0.

As a result of this analytical study, two recommendations were made with respect to the ASTM E424-71. Those two recommendations are discussed in section 2.1.3.

2.2.4 Analytical Modeling

The National Bureau of Standards has conducted studies [8, 5] on the performance and long-term durability of various solar absorber materials to aid in the development of consensus evaluation standards. The studies have shown that, even though the total integrated values of absorptance and emittance of the coating systems may exhibit little change following exposure to environmental conditions (such as elevated temperature), changes in optical properties can often be identified by comparing the reflectance spectra for aged and unaged specimens. In the absence of accurate predictive models, NBS initiated a study [12] to develop a model for determining the effect of exposure to elevated temperatures on optical properties of black chrome. First, an experimental program was conducted to find how the reflectance of the specimens varies with time of exposure. Samples consisting of three specimens of black chrome on steel substrate were exposed to elevated temperatures of 150°, 200°, and 250°C for increasing time durations. Reflectance was measured

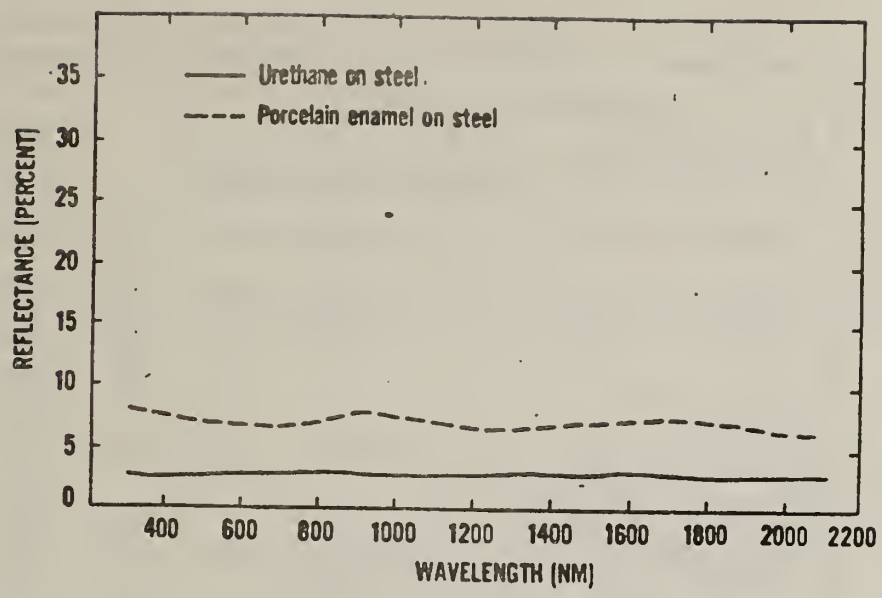

Figure 2.5 Spectral Reflectance of Nonselective Absorber Materials, Urethane on Steel and Porcelain Enamel on Steel (from reference [5])

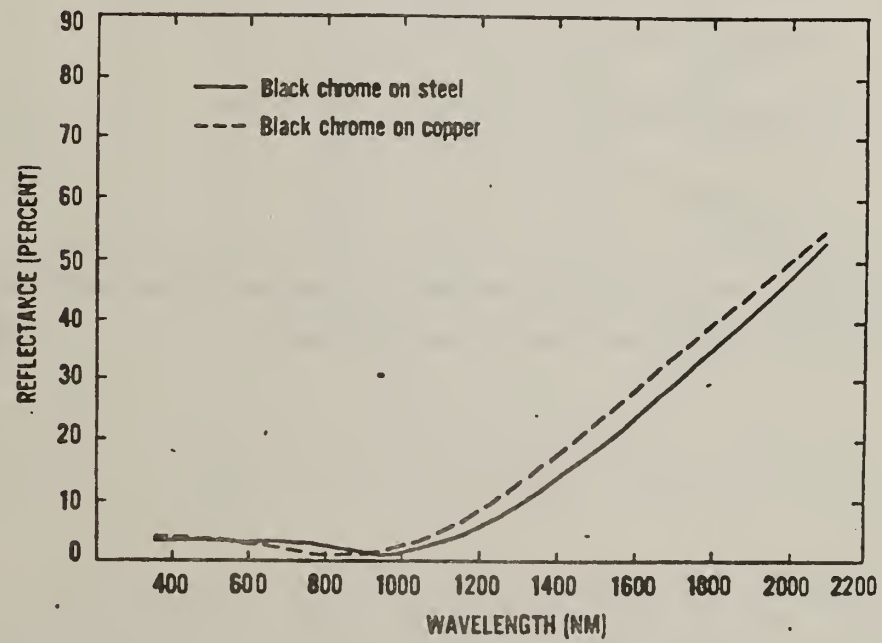

Figure 2.6 Spectral Reflectance of Selective Absorber Materials, Black Chrome on Steel and Black Chrome on Copper (from reference [5])

Table 2.12 Tabulation of Reflectance Data for Absorber Materials (from reference [5])

Material	Percent Reflectance				
	Exposure[1/] Test; Length	Weighted Ordinate at Air Mass 2.0	Selected Ord. at Air Mass 2.0	Selected Ord. at Air Mass 1.5	Selected Ord. at Air Mass 1.0
Black chrome on steel	Moisture Test[2/]	4.09	6.0	6.0	6.0
Black chrome on steel	Control	4.9	4.9	6.2	6.0
Black chrome on copper	Control	4.8	4.9	6.2	6.0
Urethane paint on steel	Control	2.9	2.8	2.8	2.8
Porcelain on steel	Control	6.6	6.5	6.6	6.6
Black chrome on steel	Moisture Test[2/] 2 weeks	4.5	4.5	5.5	5.4
Black chrome on steel	Mositure Test[2/] 6 weeks	4.2	4.2	5.3	5.2
Black chrome on steel	Moisture Test[2/] 12 weeks	3.7	3.8	4.8	4.7
Black chrome on steel	Heat Age 200°C 12 weeks	3.7	3.7	4.7	4.6
Black chrome on copper	Heat Age 200°C 12 weeks	7.0	7.4	8.6	8.2
Urethane on steel	Moisture Test[2/] 2 weeks	3.0	2.8	3.0	2.9
Urethane on steel	Moisture Test[2/] 6 weeks	4.5	4.3	4.2	4.2
Urethane on steel	Moisture Test[2/] 12 weeks	4.8	4.6	4.6	4.6
Porcelain on steel	Heat Age 250°C 6 weeks	4.3	4.3	4.2	4.2

1/ Denotes that the weathering tests were carried out prior to obtaining optical measurements.

2/ Moisture test was conducted at 92°C and 97 percent relative humidity.

Table 2.13 Qualitative Results of Parametric Study of Absorber Materials (from reference [5])

Absorber Materials		Effect on Reflectance of: Integration Methods	Effect on Reflectance of: Air Mass
	Nonselective	No difference* No difference*	No difference* No difference*
	Selective	No difference*	Higher values of reflectance with lower air mass*

* Applies to both weathered and unweathered specimens.

after 1, 10, and 22 days using an integrating sphere reflectometer as described in ASTM E424, Method A [4], before and after the exposure to the various temperature conditions. Some tests were also conducted on black chrome with copper substrate. The results of the latter test series were similar to those for the black chrome on steel specimens.

The test data indicated: 1) that the change in reflectance, at the above-mentioned temperature levels, increased with time up to 10 days, with a negligible change thereafter; and 2) that the reflectance curve (reflectance vs wavelength) undergoes a permanent horizontal shift with increasing time of exposure to the elevated temperatures. An analytical model was developed based on this horizontal shift, and a mathematical expression was derived for computing the reflectance at a specific wavelength for black chrome on metal exposed to temperatures below 250°C. Due to the limited application of the model, additional research is needed to extend the model to other absorber materials.

2.2.5 Laboratory Evaluation Devices

Two researchers at the NBS, M.A. Post and D. Ballard [13], studied the degradation of absorptive coatings by use of scanning electron microscopy (SEM) and ASTM standard test methods. The objective of the study was two-fold: 1) to assess the applicability of scanning electron microscopy for discriminating the stages of the degradation process; and 2) to obtain test data on the durability of selected absorptive coatings that could aid the development of durability-related test methods.

The three coating systems included in the study were: 1) urethane topcoat over epoxy primer on zinc-phosphate-treated steel, 2) polyvinylidene fluoride over epoxy primer on galvanized steel, and 3) epoxy topcoat over phenolic butyrate primer on anodized aluminum. The first system was exposed to condensing moisture in accordance with ASTM Standard D2247 for 14 days and was also subjected to adhesion tests before and after exposure. The other two systems were exposed to high relative humidity ($\geq$ 95 percent) and elevated temperature (92°C) for 21 weeks. Adhesion tests were not performed on coating systems 2 and 3. SEM photographs were taken of all unexposed and exposed surfaces for comparative purposes and to assist in the interpretation of microstructure.

Based on the SEM photographs obtained in the study, as compared with the standard ASTM E424 test results, it was concluded that SEM is a valuable tool for studying degradation of absorptive coatings. Some specific conclusions were drawn with respect to the degradation of the three coating systems:

1. Coating system 1 (urethane topcoat over epoxy primer on zinc-phosphate-treated steel):

    - Prolonged exposure to moisture contributes to degradation of the coating.

    - Loss of adhesion occurs in the epoxy primer with some failure in the zinc phosphate.

- The adhesion test caused only partial removal of zinc phosphate and of the urethane coating.

- Exposure to moisture and elevated temperature alters the topography of the urethane topcoat.

2) Coating System 2 (polyvinylidene fluoride over epoxy primer on galvanized steel):

- Moisture degraded the polyvinylidene fluoride coating.

- Failure of the coating may start with the absorption of moisture through holes in the mound-like areas that formed on the coating (identified by the SEM).

- The moisture penetrated to the galvanized surface, resulting in failure of the interface between the galvanized surface and the coating and the galvanized surface and the steel substrate.

3) Coating System 3 (epoxy topcoat over phenolic butyrate primer on anodized aluminum):

- Moisture degraded the epoxy topcoat.

- This degradation caused debonding between the epoxy binder and the carbon-black spheres which comprised the top coating.

- Moisture penetrated through the cracks that formed in the coating to the aluminum substrate surface, and the alumimum substrate corroded.

Most of the conclusions presented above were drawn as a result of analyzing the SEM photographs and correlating them with the physical test results.

2.2.6 Evaluation of Selective Coating Candidates

The following noteworthy studies were conducted under the auspices of the DoE, but were not included in the DoE Active Heating and Cooling Program. Because the studies are pertinent to the subject of solar absorber coatings, brief synopses are presented here.

In 1975 G.E. MacDonald of the NASA-Lewis Research Center reported [14] that black chrome had been found to have high solar absorptance in the visible region of the solar spectrum and low emissitivity in the infrared region, thus making it a promising candidate for solar absorber coating applications.

In 1976 R.B. Pettit and R.R. Sowell of Sandia Laboratories reported [15] on a study in which solar absorptance and total hemispherical emittance properties were obtained for three coatings: 1) a semiconductor-pigmented paint, 2) electroplated black-nickel, and 3) electrodeposited black-chrome. In addition to measuring the as-plated optical properties, the investigators obtained results from temperature aging and ultraviolet (UV) radiation exposure to determine the

stability of the coatings after being subjected to aging environments. Of the three coating candidates, the black-chrome was found to have the best solar selectivity properties and to be stable in high temperature and UV radiation environments.

Later, Pettit and Sowell further investigated the thermal stability of electrodeposited black-chrome as a function of various plating parameters [16]. The investigators sought to determine the composition of the plating bath so that the effect of the bath constituents on the optical properties and thermal stability of the coatings could be systematically investigated. In addition, the effects of such variables as type of substrate, current density and bath temperature on the thermal stability of the coating were evaluated. Thermal stability was determined by measuring spectral hemispherical reflectance and normal total emittance before and after thermal aging tests.

In 1979, C.M. Lampert and J. Washburn of Lawrence Berkeley Laboratory reported [17] on a study of the microstructure of a proprietary black-chrome coating to gain a better understanding of the mechanism of wavelength selectivity. The black-chrome coating was deposited on copper, steel and nickel-plated substrates. The study was conducted in three parts: 1) measuring spectral reflectance of the various coating/substrate combinations, 2) obtaining surface morphologies by scanning electron microscopy (SEM), and 3) obtaining surface morphologies by transmission electron microscopy (TEM).

In a study [18] closely related to the aforementioned one, Lampert and Washburn investigated the optical and microstructural stability of the same black-chrome coating at different temperatures and atmospheres. One-hour heat treatments were employed for temperatures ranging from the as-plated temperature to 600°C. Samples were annealed in dry air at one atmosphere and in a vacuum. To evaluate the effects of heat treatment and the annealing environment, the samples were analyzed by three procedures: 1) hemispherical spectral reflectance measurements, 2) surface and cross sectional morphologies as produced by SEM, and 3) morphologies as produced by TEM.

Although most of the research on selective coatings for flat-plate collectors has been on the deposition of black oxide coatings on low-emissivity metal substrates, some researchers have investigated alternative coating/substrate combinations. One such study was reported on in 1976 by D.P. Grimmer, K.C. Herr and W.J.Mc Creary [19] of the Los Alamos Scientific Laboratory. The objective of the study was to assess the feasibiity of producing a selective absorber by the chemical vapor deposition (CVD) of nickel dendrites on an aluminum substrate. The CVD of nickel from nickel carbonyl was thought to have the possibility of satisfying the optical requirements (i.e., high absorptance and low infrared emittance) while being cost-competitive with oxide coatings. While the test results did suggest that additional developmental research is necessary, it was concluded that the nickel dendrite coating offers a promising, low-cost technique for a selective photothermal absorber.

2.3 INSULATION

2.3.1 Introduction

Insulation, as it applies to flat plate solar collectors, serves to minimize heat loss from the back and sides of the collector enclosure. Therefore, the effectiveness of the insulation is of great importance in determining the overall thermal efficiency of the collector. Insulation should have: low thermal conductivity, dimensional stability, thermal stability under normal operating temperatures, and chemical compatibility with its environment for the life of the collector.

The greatest performance problems of collector insulation, obtained from field surveys, are: 1) moisture, 2) swelling as a result of elevated temperatures, 3) degradation due to ultraviolet radiation, and 4) condensation of volatile components on the underside of the cover plate thereby reducing transmittance and collector efficiencies.

2.3.2 Insulation Materials Research

In 1978, the National Bureau of Standards conducted a laboratory-based study [20] to develop test methods for evaluating solar collector insulation. The objectives of the study were: 1) to identify performance requirements for insulations used in solar collectors; 2) to identify or develop test methods to measure insulation performance according to these requirements; 3) to evaluate and then recommend those test methods which are suitable for the insulation materials that are used in solar collectors; and 4) to draft a set of proposed test methods for solar collector insulation materials. These methods were submitted for the consideration of the American Society for Testing and Materials (ASTM), and served as a basis for ASTM E861-82, Standard Practice for Evaluating Thermal Insulation Materials for Use in Solar Collectors.

During the study 21 types of insulation were tested. They are generally classified under four generic types: (1) mineral fibrous materials such as glass, rock or slag wool; (2) mineral cellular materials such as calcium silicate, foamed glass, perlite and vermiculite; (3) organic fibrous materials such as wood, paper or synthetic fibers; (4) organic cellular materials such as urea-formaldehyde, polystyrene or polyurethane foams. Also, insulation is generally available in four basic shapes or forms: (1) loose-fill insulation (fibers, granules or nodules) which are usually poured or blown into place; (2) flexible insulation (blanket, batt and felt) having varying degrees of compressibility and flexibility; (3) rigid insulation (preformed block, board or sheet) manufactured to standard lengths, widths and thicknesses; and (4) formed-in-place insulation (liquid components) which may be poured, frothed or sprayed in place to form rigid or semirigid foam insulation.

The following performance conditions were considered during the study: 1) sustained high or low temperatures (as high as 260°C and as low as -40°C); 2) diurnal temperature-humidity cycling; 3) condensation or accumulation of water within the collector; 4) continuous contact between the insulation and the components of the collector system; 5) mold; and 6) vibration.

The key physical properties were identified to be: mass, size, density, water absorption, moisture adsorption, thermal conductivity, and friability.

ASTM standard test methods were used in the investigation where applicable. These ASTM standards included:

C 167	Test for Thickness and Density of Blanket- or Batt-Type Thermal Insulating Materials.
C 209	Testing Insulation Board (Cellulosic Fiber), Structural and Decorative [Water Absorption].
C 553	Specification for Mineral Fiber Blanket and Felt Insulation (Industrial Type) [Moisture Adsorption].
C 518	Test for Steady-State Thermal Transmission Properties by Means of Heat Flow Meter.
C 411	Test for Hot-Surface Performance of High-Temperature Thermal Insulation.
C 356	Test for Linear Shrinkage of Preformed High-Temperature Thermal Insulation Subjected to Soaking Heat.
D 3273	Test for Resistance to Growth of Mold on the Surface of Interior Coatings in an Environmental Chamber.

Because of the absence of suitable standard test procedures, tests were developed: 1) to observe the effects of thermal cycling and outgassing; 2) to measure friability of the insulation; and 3) to observe the extent of corrosion sustained by metal parts typically in contact with insulation.

The test results, which were reported in tabular form, showed the effect of operating conditions on the insulation and on the collector component (e.g. cover plate). Tables 2.14 and 2.15 are typical of the tabulated test results.

Based on the laboratory test results a methodology was derived for screening solar collector insulation materials with respect to performance and stability under service conditions. The proposed procedures are either identical with existing ASTM tests or were developed during the study, as indicated:

Water Absorption - ASTM C 209
Thermal Conductivity - ASTM C 518
Linear Shrinkage - ASTM C 356
Corrosion - Newly developed
Hot Surface Performance - ASTM C 411
Outgassing - Newly developed
Thermal Cycling - Newly developed

Table 2.14 Effect of Hot Surface Contact on the Appearance and Linear Shrinkage of Insulations (from reference [20])

Generic Type	Sample Numbers	Surface Temperature at Which First Observable Change Takes Place[a]	Linear Shrinkage[b] at 150 and/or 260°C
Mineral Fibrous	1-FG, 2-FG 3-FG, 5-FG	209°C - Discolors and chars	None
	8-SW, 10-RW	209°C - Discolors	None
	4-FG	150°C - Discolors and chars	None
	6-FG,7-FG,9-SW	None	
Mineral Cellular	12-GF	None	None
	11-CS	209°C - Evolves water	None
Organic Fibrous	13-CL	209°C - Evolves water	c
	14-CL	94°C - Evolves water	c
	15-WF	94°C - Discolors	3% at 150°C >95% at 260°C
Organic Cellular	20-PU	150°C - Melts[d] and chars	18% at 260°C
	17-PS,18-PS 19-PS	150°C - Melts[e]	>95% at 150°C
	21-RB	94°C - Discolors	18% at 150°C
	16-UF	94°C - Bows	14% at 150°C >95% at 260°C

[a] All observations made after 96 hours of exposure to the hot surface; samples were exposed successively to 94, 150, 209, 260°C to determine the lowest temperature required to induce an observable change.

[b] Linear shrinkage calculated as the percent reduction in the original length of the sample.

[c] Shrinkage not applicable to cellulosic loose-fill insulations.

[d] Sample melts on side exposed to hot surface.

[e] Sample melts completely.

Key

FG - Fiber Glass
SW - Slag Wool
RW - Rock Wool
CS - Calcium Silicate
GF - Foam Glass
CL - Cellulose
WF - Wood Fiber
UF - Urea Formaldehyde
PS - Polystyrene
PU - Polyurethane
RB - Foamed Rubber

Table 2.15 Corrosion of Metals by Insulation Materials (from reference [20])

Generic Type	Sample No.	Degree of Pitting[a]			
		Stainless Steel	Brass	Copper	Aluminum
Mineral Fibrous	7-FG	Severe	Severe	Severe	V. Mild
	1-FG,4-FG,9-SW	--	Mild	Mild	--
	3-FG	Mild	--	--	--
	2-FG,6-FG,7-FG 8-FG,10-RW	--	--	--	--
Mineral Cellular	11-CS	--	--	Mild	--
	12-GF	--	--	--	--
Organic Fibrous	13-CL	Severe	--	Mild	Mild
	14-CL	Severe	Mild	Mild	Mild
	15-WF	--	--	Mild	--
Organic Cellular	20-PU	Mild	Mild	Mild	--
	16-UF	--	Mild	Mild	Mild
	17-PS,18-PS, 19-PS,21-RB	--	Mild	Mild	--

[a] Results reported as severe or mild pitting of the metal surface after 30-day contact with insulation; where no pitting occurred, no result is given.

The proposed new procedures are applicable to flexible, semi-rigid, rigid, loose-fill, and poured-in-place insulation materials. Also, the procedures are suitable for testing all types of insulation: mineral (fibrous and cellular), and organic (fibrous and cellular). The proposed procedures were submitted to ASTM Subcommittee E 44.04 on Materials Performance for its consideration, and, as mentioned above, served as a basis for ASTM E861-82.

2.4 SEALANTS AND GASKETS

2.4.1 Introduction

Various types and forms of rubber seals are used in solar energy systems to seal or weatherproof joints between adjoining materials or to separate dissimilar metallic materials. Although many standard test methods have been developed for rubber seals used in other building construction applications, none of these are entirely adequate for the high temperatures normally encountered in solar energy systems and subsystems. For example, the maximum service temperature of interior coverplates used in flat plate solar collectors where rubber seals are used, is estimated to be 150°C (302°F), whereas existing standards seldom consider temperatures exceeding 70°C (158°F). A need was identified to develop standards for rubber seals, for solar energy systems, which would perform at high temperatures for extended periods of time or withstand the degradation resulting from being in contact with liquids.

2.4.2 Sealant Materials Research

Research conducted at the NBS resulted in a report [21] on the topic of rubber seal performance and standards for their evaluation.

The objectives of this study included: (1) identification of performance requirements for rubber seals used in solar energy systems; (2) identification and assessment of existing test methods for rubber seals and modification of the existing methods or development of new methods as needed; (3) evaluation of commercially available rubber materials in the laboratory and acquisition of data needed to recommend specific test methods; and (4) preparation of draft standards for rubber seals for consideration as a consensus standard by the American Society for Testing and Materials (ASTM).

The key properties of rubber seals which were measured in this study included: 1) ultimate elongation; 2) compression set; 3) hardness; 4) tensile strength; 5) low temperature flexibility; 6) volatile content; 7) condensible volatiles; and 8) adhesion (for liquid seals). The key degradation factors considered were: 1) elevated temperatures; 2) low temperatures, 3) contact with liquids; 4) ozone; 5) ultraviolet radiation; and 6) stresses resulting from cyclic joint movement.

The types of rubber seal materials investigated (and their ASTM D1418* designations) included: 1) acrylate copolymer (ACM), 2) ethylene-vinyl acetate

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* ASTM D1418-79a, Recommended Practice for Rubber and Rubber Latices--Nomenclature.

copolymer (EAM), 3) chloro-sulfonyl-polyethylene (CSM), 4) fluoro rubber (FKM), 5) terpolymer of ethylene, propylene and a diene (EPDM), 6) copolymer of ethylene oxide and chloromethyl oxirane (epichlorohydrin) (ECO), 7) polychloromethyl oxirane (CO), 8) silicone copolymer containing methyl and vinyl groups (VMQ), 9) polysulfide (EOT), and 10) polyurethane (EU). Thirteen of the 31 samples were supplied and tested as vulcanized sheets. Two samples which were supplied as liquid-applied seals were prepared as sheets before testing.

The following tests for rubber were conducted on the vulcanized sheets:

ASTM D395	Test for Rubber Property - Compression Set
ASTM D412	Test for Rubber Properties in Tension
ASTM D471	Test for Rubber Property - Effect of Liquids
ASTM D865	Test for Rubber Deterioration by Heating in air (Test Tube Enclosure)
ASTM D1149	Test for Rubber Deterioration - Surface Ozone Cracking in a Chamber (Flat Specimen)
ASTM D1229	Test for Rubber Property - Compression Set at Low Temperatures
ASTM D1415	Test for Rubber Property - International Hardness
ASTM D2137	Test for Rubber Property - Brittleness Point of Flexible Polymers and Coated Fabrics

In addition, the total volatiles at 150°C and volatiles condensible at 23°C were determined by a modification of ASTM D865. Also, liquid immersion tests were performed for a period of seven days, using a modification of ASTM D471.

Sheet samples were immersed in the following liquids: 1) ethylene glycol/water (1:1); 2) propylene glycol/water (1:1); 3) polyalkylene glycol ether; 4) modified ester; 5) polyaromatic; 6) modified terphenyl; 7) silicone A; 8) silicone B; 9) silicone C; and 10) alkyated aromatic.

The test methods used for evaluating the remaining 16 liquid applied sealants included:

ASTM C792	Test for Effects of Heat Aging on Weight Loss, Cracking, and Chalking of Elastomeric Sealants
ASTM C661	Test for Indentation Hardness of Elastomeric - Type Sealants by Means of Durometer
ASTM C719	Test for Adhesion and Cohesion of Elastomeric Joint Sealants Under Cyclic Movement
ASTM C793	Test for Effects of Accelerated Weathering on Elastomeric Joint Sealants

Test results were presented in tabular form, showing the effect of specific test conditions on the samples. Table 2.16 shows a typical set of results for nine of the 11 sealant materials mentioned above. Based upon the results of the laboratory test program, five proposed standards on rubber seals in solar systems were prepared for consideration by ASTM Committee E44. The proposed standards were entitled:

Table 2.16 Results of Tests of Samples 1 through 15[1/] (from reference [21])

Sample Number	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
ASTM Designation	ACM	EAM	CSM	FKM	EPDM	EPDM	EPDM	EPDM	ECO	CO	VMQ	VMQ	VMQ	VMQ	EOT
HARDNESS - IRHD															
Original	76	57	60	74	63	60	62	65-69	72	77	47	49	26	7	44
70 h @ 150°C	76	56	68	74	68	63	64	65	82	87	46	51	30	7	melted
166 h @ 150°C	76	56	79	74	70	66	64	71	81	91	48	49	30	11	melted
TENSILE STRENGTH - MPa															
Original	8.9	15.8	18.9	12.9	8.2	12.8	14.0	10.5	12.3	14.5	7.6	9.4	1.8	0.6	1.1
70 h @ 150°C	8.8	16.8	12.2	12.1	7.6	11.6	14.4	11.4	11.1	8.2	7.6	10.6	2.0	0.6	melted
166 h @ 150°C	8.4	17.2	6.7	13.2	7.6	9.1	14.4	10.4	7.9	15.4	8.2	11.0	2.2	0.5	melted
ULTIMATE ELONGATION - PERCENT															
Original	115	380	230	205	695	390	295	540	355	280	445	660	585	285	145
70 h @ 150°C	115	370	135	185	360	225	300	405	225	165	445	670	535	205	melted
166 h @ 150°C	90	380	75	175	300	155	285	270	245	150	460	660	565	165	melted
COMPRESSION SET - PERCENT															
166 h @ -10°C	46	53	77	53	29	41	37	91	10	18	9	12	20	53	9
70 h @ 150°C	20	19	68	8	100	73	15	82	54	49	11	15	100	100	100
VOLATILES - PERCENT TOTAL															
70 h @ 150°C	0.30	0.37	1.47	2.53	0.39	0.62	0.84	0.69	1.25	0.65	0.62	0.10	0.75	0.24	—
166 h @ 150°C	0.36	0.49	2.84	2.66	0.45	0.71	0.98	0.71	1.45	0.75	0.80	0.04	0.95	0.19	—
CONDENSIBLE AT 23°C															
70 h @ 150°C	0.06	0.04	0.12	0.04	0.03	0.12	0.04	0.61	0.06	0.07	0.06	0.03	0.28	—	—
166 h @ 150°C	0.04	0.07	0.16	0.02	0.08	0.23	0.05	0.56	0.11	0.04	0.11	0.04	0.22	—	—
LOW TEMPERATURE FLEXIBILITY															
Below °C	-30	-40	-40	-15	-40	-40	-40	-40	-40	-30	-40	-40	-40	-40	-30
OZONE RESISTANCE															
166 h @ 40°C & 100 mPa O_3	← PASSED →														Failed

1/ Note retest of compression set of samples 5, 8, 9, 10 and 14 in table 5 after post-vulcanization.

1. Standard Specification for Rubber Seals Used in Flat-Solar Collectors.

2. Standard Specification for Rubber Seals Contacting Liquids in Solar Energy Systems.

3. Standard Specification for Rubber Seals Used in Solar Collectors (other than Flat-Plate Collectors)

4. Standard Test Method for Determining the Effect of Outgassing on the Transmittance of Solar Collector Covers.

5. Recommendations for Sealing Joints between Solar Collectors and Building Components with Rubber Seals.

The first three proposed standards have been adopted by ASTM as D3667-78, D3832-79 and D3771-79, respectively.

3. TRANSPORT AND STORAGE SYSTEM COMPONENTS

3.1 CONTAINMENT MATERIALS AND HEAT TRANSFER FLUIDS

3.1.1 Introduction

Among the high priority standard test method needs identified in reference [1] was a method for evaluating the durability and reliability of containment materials and heat transfer fluids in operational solar energy systems. Interaction between the heat transfer fluid and the surrounding materials may alter the properties of the fluid and have a deleterious effect on the physical properties of the containment materials. Thus, a reliable laboratory procedure for the screening of metallic and nonmetallic containment materials for possible use with heat transfer fluids was also identified as a standards need. Durability factors to be evaluated by the screening procedure included material compatibility and chemical and thermal stability of the fluids.

In addition to the need for a screening procedure, a need for comparing the corrosion resistance of various metals in operational solar heating and cooling systems during both flow and stagnation (nonflow) conditions was identified. The report noted that these simulated service tests should include such variables as flow rate, heat transfer rate, alternating wetting and drying, the degree of aeration of the transfer fluid, and the presence of crevices.

Since 1977, the objectives of several DoE-sponsored research programs have included the development or assessment of draft standards to be used in evaluating containment material/heat transfer liquid combinations for their durability and reliability when incorporated in solar energy systems. Summaries of these research programs are presented in the next paragraph. They describe the primary objectives, scope, key results, and principal conclusions obtained from the referenced reports.

3.1.2 Screening Tests for Metal/Heat Transfer Fluid Combinations

During 1978 and 1979, several national laboratories, including NBS, collaborated in a round-robin test program to assess the applicability of a proposed ASTM Standard Guide for screening metallic containment materials using metal/fluid combinations likely to be used in solar energy heating and cooling systems. The proposed standard was duly considered by ASTM Subcommittee E44.04 on Performance of Materials and served as a basis for ASTM E712-80.

In the part of the test program that was conducted by NBS [22], alloys of copper, aluminum, stainless steel, and carbon steel, in combination with ASTM D1384 tap water were subjected to five of the six tests described in the Standard Guide. The five tests which were all conducted at atmospheric pressure, were as follows: 1) basic immersion test; 2) heat rejecting surface test; 3) repeated drip dry test; 4) crevice test; and 5) tube loop test. It was felt that these five tests were adequate for simulating the effects of the following four principal system-operating conditions: 1) flowing liquid in a full system; 2) stagnant (no flow), full system; 3) stagnant, partially full system; and 4) stagnant, empty system. The metallic specimens were exposed to the test

conditions for thirty days. At the conclusion of each test, the specimens were removed, cleaned according to ASTM G1-72 Standard procedure, and weighed to determine the loss of material due to corrosion.

Upon completing the test series, it was concluded that the basic immersion test (Test #1) was effective in separating the corrosion rates of the aluminum and copper alloys. It was also determined that the corrosion rates of stainless steel alloy (AISI 409, 439 and 444) were negligible, and the variability in corrosion rates for the SAE 1014 mild steel was low. It was further concluded that Test #1 was adequate for prescreening. The results of Tests 2, 3, and 4 indicated that the variation in corrosion rate as a function of alloy composition may be determined through the use of these tests. However, Test 4 resulted in an undesirable variability in the pitting of the aluminum specimens. The test procedure was modified to reduce the variability. Test 5, while qualitative in nature, resulted in light pitting in the sample of 3003 aluminum tubing.

3.1.3 Screening Tests for Plastic Materials/Heat Transfer Fluid Combinations

Plastics can be used in place of metals as containment materials in active solar heating and cooling systems. The fact that the mechanical properties of most plastic materials are time- and temperature-dependent suggests that durability/reliability test methods are needed to assist in evaluating the long-term performance of such materials. Under operating conditions, the containment materials will be in contact with transfer fluids of varying temperatures and pressures. During stagnation conditions, components such as absorbers, transport piping and storage tanks may be subjected to elevated temperatures for sustained periods. Thus, it was necessary to develop standard test methods for screening plastic materials for their compatibility with transfer fluids such as water and glycol solutions at high temperatures.

As a consequence, the NBS engaged in a laboratory-based program [23] to obtain test data which could provide the technical basis for a screening methodology for plastic containment materials. The performance requirements and degradation factors that may affect plastic containment materials were first identified. The three general containment applications identified for plastics were: absorbers, transport piping, and storage facilities. These components should resist deterioration resulting from elevated temperatures, thermal cycling, moisture, air pollutants, and heat transfer fluids. Also, absorbers unprotected by insulation must be UV stable when subjected to direct solar radiation. Furthermore, the plastic materials must be compatible with the heat transfer fluids to the extent that they do not degrade the fluids. In the NBS study, tests were performed on 11 plastic containment materials in order to assess: 1) the effects of heat aging, and 2) their chemical compatibility with heat transfer fluids.

The containment materials included in the laboratory study are listed in table 3.1.

In the heat stability aging tests, small coupon test specimens were exposed to temperatures of 100°C (212°F) and 125°C (232°F) in a mechanical convection oven. Separate sets of specimens were used for each temperature. The materials

Table 3.1 Plastic Containment Materials Included in the Laboratory Study (from reference [23])

Materials	Description
Collectors - Absorbers	
Ethylene propylene diene monomer (EPDM)	extrusion with 1.9 mm wall
Silicone coated fiberglass fabric	0.36 mm sheet
Crosslinked polyethylene (XLPE)	17 mm diameter tubing with 1.5 mm wall
Polypropylene	extrusion, with 0.5 mm wall
Polypropylene copolymer	extrusion, with 0.64 mm wall
Piping	
Chlorinated poly(vinyl chloride) (CPVC)	34 mm OD, 4.5 mm wall
Poly(vinyl chloride) (PVC)	34 mm OD, 4.5 mm wall
Acrylonitrile-butadiene-styrene (ABS)	29 mm OD, 2.2 mm wall
Storage	
Poly(vinyl chloride) liner	0.8 mm sheet
Chlorosulfonated polyethylene liner - woven fabric	0.8 mm sheet
Fiber reinforced plastic tank	1.3 mm sheet

were evaluated for linear dimensions, hardness, weight change, appearance, and reflectance (absorbers only) by ASTM standard procedures, after 100, 250, 500, and 1,000 hours exposure. In the chemical compatibility tests, the plastic coupons were evaluated using the procedure described in ASTM D543, Test for Resistance of Plastics to Chemical Reagents. The specimens were immersed in the heat transfer fluid which was maintained at either room temperature or 70°C (158°F). The heat transfer fluids were: 1) water, 2) 100 percent ethylene glycol, 3) 50 percent ethylene glycol in water, 4) 100 percent propylene glycol, 5) 50 percent propylene glycol in water, and 6) 100 percent silicone oil. After one week of immersion the specimens were evaluated for linear dimensions, hardness, weight change, appearance, and reflectance.

As a result of this test program, it was concluded that accelerated laboratory screening tests (i.e., elevated temperature and contact with heat transfer fluids) can be effectively used to determine whether plastic containment candidates are likely to be degraded in the range of in-service temperatures of solar systems. Exposure to the six heat transfer fluids demonstrated that there are interactions between the plastic and fluids in the test temperature range. Silicone oil generally caused the plastics to swell, soften, and gain weight. The effects of the other fluids ranged from essentially no change to an increase in hardness or softness, weight gains or dimensional changes.

Based upon the NBS laboratory study, a draft standard was prepared and submitted to ASTM Committee E44 on Solar Energy Conversion. Subsequently, ASTM adopted a new national standard, E 862-82 [24] which was based on the draft standard resulting from the above mentioned study.

3.1.4 Simulated Service Testing for Corrosion

Because the interaction between the heat transfer fluid and its containment system can result in deleterious changes in the properties of the fluid and/or corrosion of metallic components of the containment system, it is often advisable to evaluate the responses of containment systems under actual or simulated operating conditions. The NBS participated in a study [25] to assess the validity of a proposed ASTM test method in which accelerated metallic corrosion is induced during simulated flow and stagnation conditions in a solar collector system. The corrosion responses of aluminum, copper and stainless steel tubing, and the chemical stabilities of ethylene and propylene glycol solutions were evaluated during the study.

In the heat transfer liquid stability test series, coupons of 1100 aluminum and 110 copper alloys were immersed in solutions of ethylene glycol and propylene glycol at 100°C (212°F) for 16 weeks. The solutions were either aerated with compressed air or deaerated with high purity nitrogen. Based on the assumption that the degradation of ethylene and propylene glycol results in the generation of an acid solution, pH was measured against time to determine the degree of degradation. The results of this test series were presented as graphic plots of pH vs time for three sets of conditions: 1) aeration with no metal present, 2) deaeration in the presence of aluminum and copper coupons at 100°C, and 3) aeration in the presence of aluminum and copper coupons. It was concluded that the presence of copper metal and the availability of oxygen accelerated the degradation of ethylene and propylene glycol, while the presence of aluminum had a minimal effect. It was also observed that while ethylene glycol slightly more thermally stable than propylene glycol, it was significantly less oxidatively stable. Because of the latter observation, ethylene glycol was generally used in the series of seven simulated service tests. Distilled water only, containing 1,000 ppm of NaCl was used in one test.

Four of the simulated service tests were conducted using aluminum tubing, two tests used copper tubing and one test involved stainless steel tubing. The systems were operated under conditions of either continuous flow at 90°C, or cycles of flow followed by stagnation at 90°, 120°, and 135°C. A flow rate of 0.64 m/s was used in all systems. Test durations were 16 weeks and 24 weeks.

In the continuous circulation tests, the transfer liquid was pumped from a heated reservoir through a system of tube loops simulating a typical collector. A schematic of the various components of the loop system is shown in figure 3.1. In the stagnation phase, the liquid was allowed to stand in one of the vertical legs for 4 hours, while the other vertical leg remained empty. The corrosion rate of the metallic tube specimen was determined by measuring mass loss for each of the 7 tests. The test conditions and test results are summarized in tables 3.2 and 3.3.

It was observed that the corrosion rates in both copper systems were low, although the corrosion rate on the stagnant empty side of Copper 2 was higher than on the stagnant full side. The stainless steel test was inconclusive although some mass loss and pitting were observed. Because the four aluminum systems contained an uninhibited, unbuffered glycol solution, the corrosion rates were higher. The corrosion rates in the stagnant empty legs for the three aluminum systems increased with increasing stagnation temperature. Since the methodology used in this study was found to be adequate for the metals and heat transfer fluids commonly used in collector systems, it was submitted for consideration by ASTM Committee E 44 (on Solar Energy Conversion), and served as the basis for ASTM E 745-80 [26].

In another laboratory-based study [27], Argonne National Laboratory evaluated the corrosion behavior of representative metals (6061 aluminum, CA 122 copper, black iron, 1008 low-carbon steel, galvanized black iron and galvanized 1008 steel) in ethylene glycol-water solutions under temperature and flow conditions common to solar collector systems. The corrosion experiments were conducted under the heat-flux conditions encountered in normal operations. Several experiments were performed in a static autoclave at elevated temperatures, which simulated stagnation conditions in the collector. The thermal stability and corrosivity of the fluids were evaluated. The feasibility of using an electrochemical sensor to instantaneously measure corrosion rates instead of using metal weight loss measurements was also investigated.

The corrosion behavior of the metal specimens was determined by using a heat-flux loop facility (HFLF) which consisted of a heat-rejection loop and a heat-absorption loop. During operation, the two loops were maintained at different temperatures while a mixing flow was maintained between the loops to simulate thermal cycling of the fluid in a solar collector system. The corrosion behavior of a metal at a low temperature was thus evaluated while in contact with fluids subjected to high temperatures in the same flow system. The heat-transfer fluids used in the corrosion study were aqueous solutions of ethylene glycol. Most of the experiments involved an inhibited ethylene glycol (IEG) solution, manufactured for use as an automotive coolant. The coolant was diluted with corrosive water (CW) prepared according to ASTM D2570. For comparative purposes, tests were also conducted with uninhibited (i.e., anti-freeze-grade) ethylene glycol (UEG), diluted with the same corrosive water solution. The metal specimens were 25.4-mm lengths of ring sections of 3/8 in Schedule 40 pipe. Several lengths formed the upper half of each specimen which served as a heat flux ring, while other lengths comprised the lower half serving as an isothermal ring. In the heat-rejection loop, the heat-flux ring was electrically heated, while in the heat-absorption loop the ring was

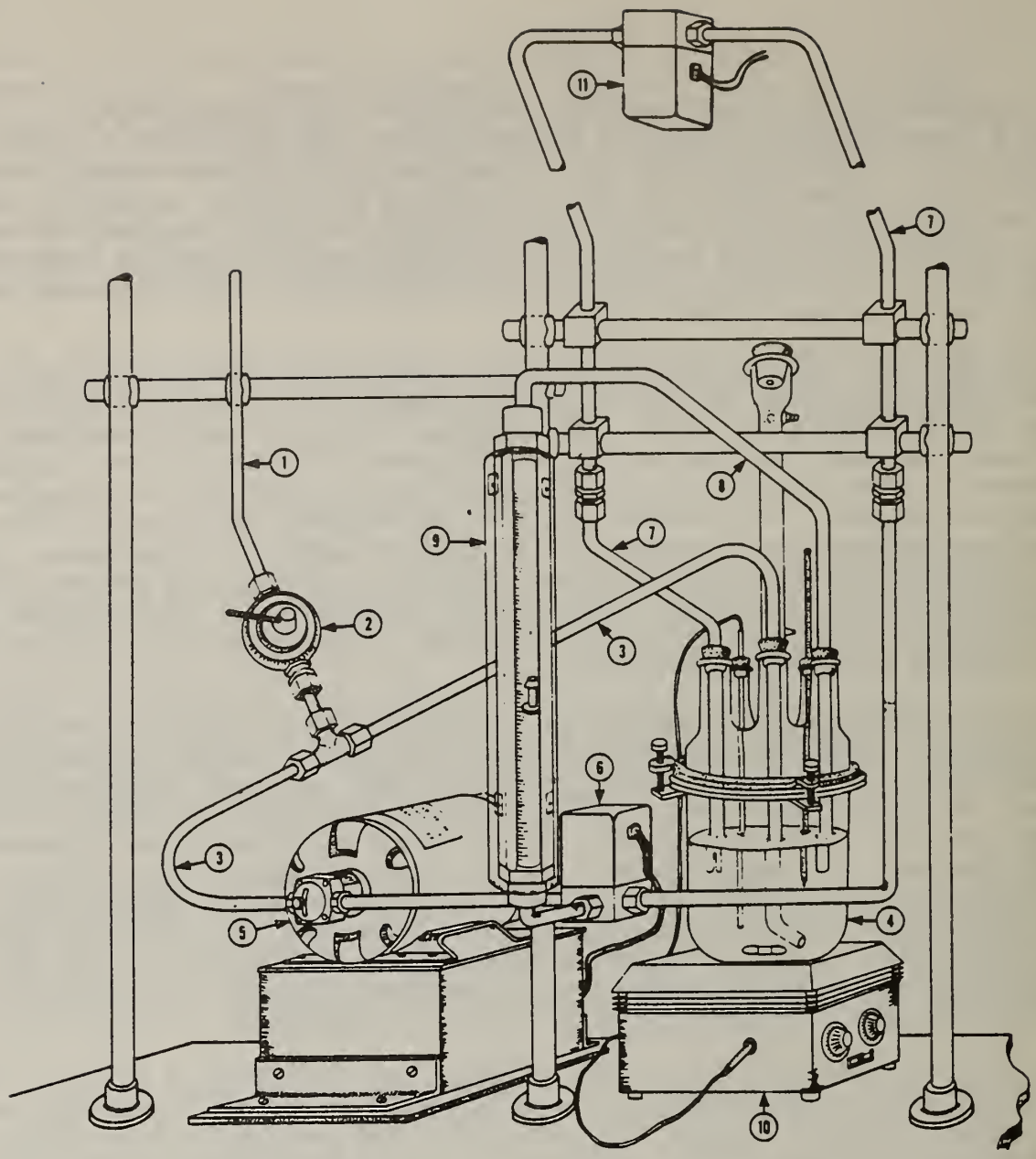

Figure 3.1 Schematic Showing the Various Components of the Loop Systems.

Systems are initially charged with liquid through the priming port [1] by opening the manual two-way valve [2]. The common flow path [3] is from the reservoir [4], through the pump [5], to the 3-way solenoid valve [6]. The primary flow path [7] is across the samples. The flow path is changed from primary to secondary by activating the 3-way solenoid valve [6] and the secondary path [8] during loop stagnation is through the flowmeter [9]. The hot plate [10] maintains the liquid temperature during stagnation. In systems which stagnate above the boiling point of the liquid, a 2-way solenoid valve [11] placed at the top of the loop is activated during stagnation. In continuous flow systems, a manual 3-way valve replaces the 3-way solenoid valve [6]. (From reference [25].)

Table 3.2 Summary of Test Conditions for Tubing Specimens (from reference [25])

Tubing Alloy	System	All Other Materials* Exposed to Each Heat Transfer Solution	Compositions of Solutions	Temperature Flow	Temperature Stagnation	Flow Rate	Test Duration
3003 AL	Al #1	Teflon, epoxy,	50% ethylene	90°C	--	0.64 m/sec	16 weeks
	Al #2	polyethylene,	glycol, 50%	90°C	90°C	"	"
	Al #3	glass, 316 stain-	ASTM D1384	90°C	120°C	"	"
	Al #4	less steel	water	90°C	135°C	"	"
122 Cu	Cu #1	Above plus 95/5	25% ethylene	90°C	--	0.64 m/sec	24 weeks
	Cu #2	Sn/Sb solder	glycol, 25% GM 6038-M, Type A antifreeze, 50% ASTM D1384 water	90°C	120°C	"	"
316 S/S	S/S #1	Teflon, epoxy, polyethylene, pyrex glass	Distilled water +1,000 ppm NaCl	90°C	120°C	0.64 m/sec	16 weeks

* Used for connectors, sealants, or as components of valves, pump, tubing, etc.

Table 3.3 Summary of Test Results (from reference [25])

Tubing System	T, °C Flow	T, °C Stagnation	pH Initial	pH Final	Corrosion Rate, mpy* Empty Side	Corrosion Rate, mpy* Full Side	Deepest Pit Empty Side	Deepest Pit Full Side
Al #1	90	--	8.35	5.56	0.49		1.06 mm	
Al #2	90	90	--	5.08	0.38	0.40	negl.	negl.
Al #3	90	120	--	5.33	0.54	0.28	0.85	0.55
Al #4	90	135	--	5.64	1.34	0.67	0.12	negl.
			Reserve Alk.					
Cu #1	90	--	3.89	3.56	0.03			
Cu #2	90	120	3.89	3.05	0.12	0.08		
			pH					
S/S	90	120	7.72	8.57	negligible			

*microns per year

(Reserve alkalinity refers to the amount of buffering remaining.)

air cooled. Only the outside surface of the specimens was in contact with the flowing ethylene glycol solutions.

In the first study conducted with the HFLF, the ring specimens were exposed to a 50 percent solution of UEG in corrosive water to establish baseline data for comparison with results from specimens exposed to the IEG solutions. The specimens were exposed to a flowing UEG solution for about 48 hours at temperatures of 140 and 105°C (413 and 378°K) in the heat-rejection and heat-absorption loops, respectively. The heat fluxes in the two loops were ~1.0 and 0.4 kW/m^2 respectively. Material weight-loss measurement was made and corrosion rates were determined. Then, two 1000-hour tests were performed in the HFLF with a 50 percent solution of IEG and corrosive water. The temperature of the fluid in the heat rejection loop was 120°C (393°K) in the first 1000-h test and 140°C in the second run; the temperature in the heat absorption loop was 105°C in both runs. The specimens were weighed to determine metal weight-loss after about 500 hours of exposure and at the end of the 1000-hour period. The specimens were observed for visible corrosion and were also photographed. The measured metal weight loss values and the corresponding corrosion rates were presented in tabular form for the three test runs. Representative results are presented in table 3.4.

In addition to the weight-loss measurement method of determining corrosion rates, two other trial methods of making direct corrosion rate measurements were employed: 1) a commercial electrochemical corrosivity sensor, and 2) potentiodynamic corrosion measurements. A commercial corrosivity sensor developed for use in automotive coolant systems was redesigned for application in solar energy systems. The sensor continuously measured the electrical potential of the solution. As the ethylene glycol degraded, its pH changed due to the formation of corrosive acids and there was a corresponding change in the potential of the sensor's probe in the solution. As the solution became more acidic, its corrosive ability increased. Thus, it was possible to correlate the sensor output (i.e., voltage) with the corrosive behavior of the metal specimens. Although the sensor could readily distinguish changes in pH, it was concluded that additional data are required before a final assessment of the sensor energy system can be made.

With regard to the potentiodynamic polarization measurements mentioned above, data were obtained on the corrosion potential, corrosion current and the instantaneous corrosion rate for iron, copper, and aluminum in ethylene glycol-corrosive water in the as-formulated condition and after exposure to a temperature of ~125°C (400°K) for 1000-hours in the HFLF.

Among the conclusions resulting from the three tests in the heat-flux loop facility were the following:

1. A heat flux of 0.4 to 1.0 kW/m^2 did not have a significant effect on the corrosion behavior of iron, copper, and aluminum in a 50 volume percent inhibited ethylene glycol-corrosive water solution at temperatures between 105 and 140°C (378 and 413°K). The corrosion rate of copper was considerably higher than that of iron or aluminum in the 1000-h tests at low flow velocities.

Table 3.4 Corrosion Results for Aluminum, Copper, and Iron After Exposure to 50 Volume Percent IEG* (GM-6038M)-CW* Solution for 1000-h in the HFLF* (Run 3) (from reference [27])

Material	Time, h	Specimen Location							
		Heat Rejection Loop (140°C)				Heat Absorption (105°C)			
		Isothermal		Heat Flux (1.0 kW/m^2)		Isothermal		Heat Flux (0.4 kW/m^2)	
		Defilmed Metal Loss, mg	Corrosion Rate,[a] mm/y	Defilmed Metal Loss, mg	Corrosion Rate,[a] mm/y	Defilmed Metal Loss, mg	Corrosion Rate,[a] mm/y	Defilmed Metal Loss, mg	Corrosion Rate,[a] mm/y
Aluminum (6061)	500[b]	2	0.010	2	0.010	c	c	c	c
	1000	3	0.007	3	0.007	c	c	c	c
Copper (CA 122)	500[b]	184	0.26	194	0.28	15	0.02	11	0.02
	500[d]	63	0.09	-	-	15	0.02	-	-
	1000	292	0.21	257	0.18	28	0.02	25	0.02
Black Iron (As-received)	500[b]	11	-	17	-	12	-	21	-
	1000	24	-	22	-	29	-	40	-
(Polished)	500[b]	6	0.010	5	0.008	7	0.011	12	0.020
	1000	6	0.005	8	0.007	11	0.009	14	0.011

[a] Surface area of the 3/8-in Schedule 40 pipe specimens was 13.8 cm^2. Value based upon linear rate law.

[b] Time corresponds to the first 500-h of the 1000-h experiment.

[c] Specimens exhibited a slight net weight gain after defilming and correcting for metal loss due to the chemical cleaning operation.

[d] Time corresponds to the last 500-h of the 1000-h experiment.

* IEG - Inhibited ethylene glycol solution

CW - ASTM D2570 corrosive water

HFLF - Heat-flux loop facility

2. In a short-term (48 hours) corrosion test at 140°C with a 50 volume percent solution of uninhibited ethylene glycol in corrosive water, aluminum exhibited localized pitting, and 70 percent of the zinc coating on galvanized iron was removed. The corrosion rate of iron was significant, whereas copper experienced minimal attack in the fluid at a low flow rate.

3.2 HOSES AND FLEXIBLE COUPLINGS

3.2.1 Introduction

Rubber hose is used extensively in solar energy systems to connect solar collectors to manifolds and to connect other components in these systems. Hose has several advantages for making these connections; for example, rubber hose does not place high stresses on the components to be connected. On the negative side, the disadvantages of rubber hose include the frequent occurrence of leakage at the connections and uncertain life expectancy when used in the severe temperature conditions common to solar energy systems. The need for developing standards that account for the advantages and disadvantages of using rubber hose was the basis for two studies [28,30] conducted by the NBS during 1979 and 1980. Although some standards exist for rubber hose used in specific industries (e.g., automotive), they are generally not suitable for hose used in solar energy systems. This inapplicability is due primarily to durability considerations under the severe environmental conditions encountered in solar systems.

3.2.2 Research on Rubber Hoses for Solar Energy Systems

The objectives of the first study [28] were: 1) to identify the performance requirements for rubber hose used in solar systems; 2) to assess existing standard test methods for rubber hose and develop new ones where needed; 3) to evaluate commercially available hose as a basis for making recommendations on standards requirements; and 4) to prepare draft standards for rubber hose for consideration by ASTM as consensus standards.

Eleven commercial rubber hoses and one polytetrafluorethylene (PTFE) hose with stainless steel fibrous braid were included in the study. These hoses were not designed for use in solar energy systems. There were three categories of hoses according to rubber type: 1) chloropene rubber (CR) (neoprene); 2) terpolymer of ethylene, propylene, and a diene (EPDM); and 3) silicone rubber having both methyl and vinyl groups on the polymer chain (VMQ).

A total of five tests were performed, the first one involving the continuous circulation of ethylene-glycol/water mixture through the twelve hoses for a period of more than seven months. The circulation tests were conducted under a predetermined temperature cycle as follows: 1) at 100°C for 82 percent of the time; 2) between -25°C and -40°C for 2 percent of the time; and 3) between 10°C and 25°C for 16 percent of the time. The remaining four tests covered: 1) ozone resistance in a chamber at 40°C; 2) compression set resulting from the pressure exerted by a prescribed compression clamp; 3) water vapor transmission loss through the hose wall; and 4) bursting pressure using a hand-operated

hydraulic pump. For determining the ozone resistance of the hoses, the procedures in ASTM D380 and D1149 were used. ASTM D395 was modified for use in measuring compression set. A new test method was devised for measuring water vapor transmission through hoses. The procedure in ASTM D380 was followed in measuring the bursting pressure, except for a minor modification.

According to the investigators, the study achieved the objective of determining the important requirements of hoses used in solar energy systems. Based on the results of the study, a proposed standard specification for rubber hose was prepared. The standard has requirements for evaluating the following characteristics: 1) physical properties of inner tube and outer cover before and after accelerated aging; 2) resistance to ozone of the outer rubber cover; 3) compatibility of the inner tube with the heat transfer fluid; 4) flexibility at low temperature; 5) compression set of the composite hose; 6) bursting pressure of the composite hose; 7) vapor transmission through the hose walls; 8) effect of the hose on heat transfer fluid and metal; and 9) durability of reinforcement material. Laboratory-based tests are included for assessing all of these characteristics, except for the deterioration of hose reinforcement material. A short-term laboratory test is not adequate to assess this characteristic. However, the behavior of fibers at various temperatures in many environments is known or could be established.

A proposed standard specification was submitted for consideration by ASTM Subcommittee D11.3 on Rubber Hose. The draft standard, with minor modifications, was adopted by ASTM as D3952-80 [29].

Because ASTM D3952-80 [29] did not cover the testing of rubber hoses used in liquids above their boiling point, a need for specifying the performance of hoses used with aqueous liquids at temperatures above the boiling point of water was identified. Thus, a second study [30] was initiated in 1980 to determine what revisions of the standard, if any, were necessary. The study consisted of conducting interlaboratory tests on commercial hose linings.

Specimens of six hose lining vulcanizates, in 2-mm-thick sheet forms were immersed in an aqueous glycol solution at both 100 and 150°C for periods of 166 h and 332 h, respectively. Since some of the tests were conducted at temperatures above the fluid's boiling point, a pressure vessel was used. The pressure vessel consisted of commercial pipe welded shut at one end and equipped with a large flange at the other to accommodate a pressure relief valve opening at 1000 kPa. In addition to the immersed specimens, there was a set of untreated control specimens whose properties served as a basis of comparison. Mass measurements were made before and after immersion to calculate the percent change. Also, the tensile strength and ultimate elongation of the immersed and the control specimens were measured to determine the change in these properties due to the immersion. Finally, the physical appearance of the specimens and the test liquid were observed before and after immersion for resulting changes.

It was concluded from the study that temperature and time of immersion are not critical for good vulcanizates. However, one of the silicone rubber vulcanizates, although complying with ASTM D3952-80, did not withstand temperatures

above 100°C, and would not be satisfactory where temperatures frequently reach 150°C. It was decided, therefore, that ASTM D3952-80 was not adequate to assure satisfactory performance of rubber hose for solar energy system use at temperatures above coolant boiling points. Consequently, proposed revisions to ASTM D3952-80 were prepared. The proposed revisions have not, as of this writing, been adopted.

3.3 STORAGE MEDIA

3.3.1 Introduction

Storage of solar energy is one of the more important aspects of the efficient utilization of solar energy. Storage of sensible heat in rock bins or water tanks has long been used, but these storage methods require considerable space. In contrast, phase change storage systems may require as little as one-seventeenth of the volume of equivalent rock storage or one-eighth of the volume of water. Another advantage in using phase change materials (PCM's) is that thermal stratification does not occur in phase change storage systems because temperatures remain nearly constant throughout the charge-discharge cycle.

In a phase change system the solar energy is converted into latent heat of fusion within the PCM. Only after the phase change is completed and all of the latent heat is absorbed, will the temperature of the PCM increase again. When the PCM is allowed to cool, the reverse process takes place; latent heat is released as sensible heat before the temperature of the PCM decreases again.

During 1980 the NBS conducted a review of the literature to identify PCM systems currently in use or with potential for widespread use, and to identify problems associated with the PCM's and their containment systems. The results of the literature review were reported in February 1982 [31]. The literature study was a forerunner to experimental work needed for developing test methods by which the performance of inorganic PCM's can be evaluated.

3.3.2 Background

PCM's used in solar applications are typically either organic materials (such as paraffin wax) or inorganic salts (such as sodium sulfate decahydrate, which is commonly called Glauber's salt). The latent heat exchange in paraffin occurs during melting and solidification of the wax at a constant temperature. For Glauber's salt the phase change occurs during formation of a saturated aqueous solution of sodium sulfate and crystalline sodium sulfate precipitate. In addition to being divided into organic and inorganic groups, PCM's are further divided into four types of encapsulation or containment: 1 & 2) macroencapsulation in metal and in plastic (e.g. trays, pouches, cans, tubes, and boxes); 3) microencapsulation (small granules or pellets of PCM packaged in individual envelopes), and 4) bulk storage in tanks. Phase change systems presently in use or with future potential are summarized in tables 3.5 and 3.6.

Table 3.5 Typical Combinations of Inorganic PCM's and Encapsulating Materials for Thermal Storage (from reference [31])

Type of Encapsulant	Inorganic System			
Plastic	Glauber's salt in rigid plastic containers. Problems with performance. [C]	Ceiling tiles filled with Glauber's salt in plastic foil pouches. [C]	Salt hydrate mixture in flexible plastic chubs. [A]	Disodium phosphate dodecahydrate in rigid plastic. Water permeability a concern. [R]
	$CaCl_2 \cdot 6H_2O$ eutectic mixture in high density polyethylene blackened tubes. [C]	Salt hydrates in rigid plastic trays. Problems with salt segregation. [A]	$CaCl_2 \cdot 6H_2O$ is high density polyethylene tubes 50-75 mm diameter. [R]	Glauber's salt in rigid polyethylene trays. [C]
Metal	Glauber's salt in stainless steel tubes. [C]			
Bulk	Glauber's salt with immiscible heat transfer liquid in tanks. [C]	Salt hydrate in tanks with heat exchanger. [A]	Sodium acetate trihydrate with intermediate immiscible heat transfer fluid. No solid heat transfer surface. [A]	Rotating drum with Glauber's salt inside with heat exchangers. [R]
	Sodium thiosulfate pentahydrate in tanks with plastic heat exchange tubes. [C]	Various salt hydrates in tanks with high density polyethylene heat exchangers. Tested 1000 cycles. [A]	Disodium phosphate decahydrate and miscible heat transfer fluid. Problems with surface. [R]	
Microencapsulation	Glauber's salt crystals coated with 3% of encapsulating material, packaging in trays. [C]			

C = commercially available A = advanced demonstration stage R = research stage

Table 3.6 Typical Combinations of Organic PCM's and Encapsulating Materials for Thermal Storage (from reference [31])

Type of Encapsulant	Organic System		
Plastic	Poroplastic sheets impregnated with stearic acid-paraffin combination. [R]		
Metal	Paraffin in 1 gal. steel cans. Tested 3 years, few problems identified. [C]	Paraffin in beer cans. [R]	Paraffin stored in modular Al panels to supply process water. [C]
Macroencapsulation	Form-stable crystalline polymer pellets (Melting Point - 120-140°C) in ethylene glycol. [R]	Form-stable, electron crosslinked polyethylene pellets. [R]	50-100 μm paraffin spheres encapsulated in polymeric materials. Used in a 40% solids-water slurry. [C]

C = commercially available A = advanced demonstration stage R = research stage

3.3.3 Problem Identification

Upon studying the literature on PCM's, the NBS investigators were able to identify degradation characteristics of inorganic and organic PCM's, and of various encapsulating materials. Three major problems associated with inorganic salt hydrate PCM's are: 1) crystal segregation due to incongruent melting of the salt hydrates, 2) supercooling to the point below which useful heat can be extracted, and 3) crystal growth rate and growth habits which can adversely affect the rate of heat transfer. The third problem becomes particularly critical when thermal cycling occurs in systems to which thickening agents have been added.

The investigators concluded that organic PCM's may be slightly less desirable than inorganic salt hydrates due to lower heats of fusion, higher cost, and the danger associated with storing large quantities of flammable substances. However, the organic PCM's do allow repeated thermal cycling with no apparent loss in thermal performance. The most promising organic systems identified consist of paraffin waxes encapsulated in either steel or plastic.

In addition to the problems associated with PCM's, there are factors which may contribute to the degradation of the encapsulating materials. Metals, except stainless steel, have not been used with salt hydrates due to the likelihood of corrosion. Instead, salt hydrates are macroencapsulated in plastic materials. On the other hand, paraffin waxes are not compatible with most plastics since the waxes can cause stress cracking in these materials. The potential problem of water migration through the plastic container walls was also considered. Water lost through the walls can not be conveniently replaced, and would in time reduce the efficiency of the system since insufficient water would be available to completely rehydrate the PCM salts.

3.3.4 Performance Test Method Needs

It was concluded from this study that PCM technology is tending toward the use of inorganic salt hydrate phase change systems. Thus, there is a need for the development of test methods by which the long-term performance of these phase change systems can be evaluated. The methods should address the phenomena of material degradation and loss of energy storage capacity. Two major obstacles to the development of performance tests are: 1) the diversity of possible materials and system designs requires that the test methods be sufficiently flexible to accomodate unanticipated materials and systems, and 2) test methods must consider the effects of both acceleration (of tests) and simulation of actual operating conditions. Acceleration is usually attainable, but simulation depends on an understanding of failure mechanisms (such as settling of salts), some of which may be unknown or poorly understood. However, test parameters for a generalized thermal cycle of phase change can be identified, including: 1) maximum temperature during the cycle, 2) transition temperature in heating, 3) transition temperature in cooling, 4) minimum temperature during the cycle, and 5) rate of change from one temperature to another.

4. SUMMARY OF ACCOMPLISHMENTS AND CURRENT STUDIES

4.1 RESEARCH ACCOMPLISHMENTS

Consistent with the priority rankings listed in table 1.1, the solar energy materials research since 1974 has largely focused on absorptive coatings and cover plates. The other high-priority areas have also received some research attention, as indicated on table 4.1. The intent of table 4.1 is to show the distribution of the studies summarized herein with respect to the various high- and medium-priority materials categories. Listed in the first column are the key properties associated with the identified materials. The bracketed numerical entries correspond to the reference numbers listed in the index of references which follows this chapter. Thus, by referring to table 4.1 and to the index of references, one can identify studies that concentrated on the evaluation of one or more key properties of a given material.

Much of the materials-related research conducted by national laboratories has been aimed at the development of standard test methods with which to evaluate: initial properties, performance under extreme operating conditions, and long-term performance. A number of new or modified standards have resulted, at least in part, from the studies summarized herein. Table 4.2 lists 18 new ASTM solar energy materials standards, all of which were adopted since 1978. Where appropriate, these standards take into account some of the conditions that occur in solar energy heating and cooling systems such as stagnation, and the corrosivity of heat-transfer fluids in contact with metallic containment materials.

4.2 FISCAL YEAR 1982 RESEARCH ACTIVITIES AT NATIONAL LABORATORIES

Several national laboratories are currently engaged in a wide range of research activities related to solar energy materials. As indicated by the projects listed in table 4.3, the primary focus is still on absorptive coatings and cover plate materials. Table 4.3 lists the current project titles, together with a statement of the primary objectives for each project. In addition to seeking to develop standard test methods, the table indicates that some projects are also aimed at developing mathematical models of various physical or chemical processes that affect solar energy materials. By using such models, key mechanisms of degradation can be mathematically modeled. It should then be possible, for example, to screen and select materials without engaging in extensive long-term testing.

Table 4.1 Distribution of Studies on Solar Energy Materials Research *

	High Priority						Medium Priority	
Material Description / Key Properties	Absorptive Coatings and Substrates	Cover Plates	Heat Transfer Fluids	Containment Materials	Collector Insulation	Sealants	Hoses & Flexible Materials	Thermal Storage Media
Optical Properties	[5][8][9][12][14] [15][16][17][18][19]	[3][5]						
Durability	[2][5][8][9][12]	[2][3][6][7]	[2][22][27]	[2][22][23] [25][27][31]	[2][20]	[2][21]	[2][28][30]	[2][25][27][31]
Resistance to Environmental Exposure	[5][8][9]	[2][3][6][7]			[20]	[2][21]	[2][28]	
High Temperature Performance	[2][5][8][12][18]	[3]	[25][27]	[25][27]	[20]	[21]	[30]	[25][27][31]
Materials Compatibility/ Corrosion	[2][8][9][13][17] [18]	[2][3]	[2][22][23] [25][27]	[2][22][23] [25][27][31]	[2][20]	[2][21]	[2][28][30]	[2][8][23][25] [27]
Resistance to Fire and Impact		[3][7][15]						

* Numbers in [ ] refer to references listed in Chapter 6.

Table 4.2 List of Standards on Solar Energy Materials Adopted By the American Society for Testing and Materials

Absorptive Coatings and Substrates

E744-80 - Standard Practice for Evaluating Solar Absorptive Materials for Thermal Applications

E781-81 - Standard Practice for Evaluating Absorptive Solar Receiver Materials When Exposed to Conditions Simulating Stagnation in Solar Collectors with Cover Plates

B638-81 - Standard Specification for Copper and Copper-Alloy Solar Heat Absorber Panels

Cover Plates

E765-80 - Standard Practice for Evaluation of Cover Materials for Flat Plate Solar Collectors

E782-81 - Standard Practice for Exposure of Cover Materials for Solar Collectors to Natural Weathering Under Conditions Simulating Operational Mode

E822-81 - Standard Practice for Determining Resistance of Solar Collector Covers to Hail by Impact with Propelled Ice Balls

E881-82 - Standard Practice for Exposure of Solar Collector Cover Plate Materials to Natural Weathering Under Conditions Simulating Stagnation Mode

Collector Insulation

E861-82 - Standard Practice for Evaluating Thermal Insulation Materials for Use in Solar Collectors

Containment Materials and Heat Transfer Fluids

E712-80 - Standard Practice for Laboratory Screening of Metallic Containment Materials for Use with Liquids in Solar Heating and Cooling Systems

E745-80 - Standard Practices for Simulated Service Testing for Corrosion of Metallic Containment Materials for Use with Heat-Transfer Fluids in Solar Heating and Cooling Systems

E862-82 - Standard Practice for Screening Polymeric Containment Materials for the Effects of Heat-Transfer Fluids in Solar Heating and Cooling Systems

Table 4.2 List of Standards on Solar Energy Materials Adopted by the American Society for Testing and Materials (continued)

Sealants

D3667-78 - Standard Specification for Rubber Seals Used in Flat Plate Solar Collectors

D3771-79 - Standard Specification for Rubber Seals Used in Concentrating Solar Collectors

D3832-79 - Standard Specification for Rubber Seals Contacting Liquids in Solar Energy Systems

D3903-80 - Standard Specification for Rubber Seals Used in Air-Heat Transport of Solar Energy Systems

Hoses/Flexible Couplings

D3952-80 - Standard Specification for Rubber Hose Used in Solar Energy Systems

General Optical Property Measurements

E434-80 - Standard Test Method for Calorimetric Determination of Hemispherical Emittance and the Ratio of Solar Absorptance to Hemispherical Emittance Using Solar Simulation

E903-82 - Standard Test Method for Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres

Table 4.3 Current Materials Research for Solar Heating and Cooling by National Laboratories

Component(s)	Project Title	Objective	Research Laboratory
Absorptive Coatings	Degradation of Absorptive Coatings	To develop mathematical models for the degradation processes of absorber materials which can be used to predict long-term durability.	National Bureau of Standards
	Mathematical Models for Degradation of Polymeric Materials	To develop mathematical models for predicting the rate at which polymeric absorber coatings degrade in solar collectors.	National Bureau of Standards
	Chemical Conversion Coatings	To investigate the feasibility of low-cost chemical coatings for use in solar collectors.	Los Alamos National Laboratory
Cover Plates	Flame Spread of Glazing Materials	To determine whether a correlation exists between ASTM D635 and actual fullscale testing, and to make appropriate recommendations to ASTM.	National Bureau of Standards
	Degradation Analysis of Cover Plates	To identify methods of detecting degradation of cover plate materials at early stages thereby providing a useful tool for screening candidate cover plate materials in the laboratory.	National Bureau of Standards
	Advanced Glazing Systems for Flat Plate Collectors	To determine the technical and economical feasibility of high performance glazing systems applied to flat-plate collectors.	Solar Energy Research Institute

Table 4.3 Current Materials Research for Solar Heating and Cooling by National Laboratories (continued)

Components(s)	Project Title	Objective	Research Laboratory
Heat Transfer Fluids/Corrosion	Kinetics of Glycol-Based Antifreeze Degradation	To develop rate equations which describe the degradation kinetics of ethylene and propylene glycol and to identify the mechanistic paths by which they degrade.	National Bureau of Standards
	Inhibitors/Buffers for Glycol-Based Heat Transfer Fluids	To investigate the utilization of common degradation product additives as a basis for decelerating corrosion attack of metallic containment systems.	National Bureau of Standards
Containment Materials	Test Methods for Nonmetallic Containment Materials	To develop draft test methods for nonmetallic materials used in solar energy systems.	National Bureau of Standards
Desiccants	Development of Improved Desiccants	To determine the feasibility of tailoring absorptive properties with high capacity and low heats of absorption for use in improved desiccant cooling systems.	Argonne National Laboratory
Phase Change Materials	Test Methods for Phase Change Materials (PCM's)	To provide assessment of the long-term performance and durability and to identify failure mechanisms of PCM's.	National Bureau of Standards

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11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

A summary of the solar energy materials-related research projects conducted by the National Bureau of Standards, for the U.S. Department of Energy, since 1975 is presented. Research studies concerned with materials that are utilized in the collector, transport and storage subsystems are summarized. Materials research areas covered by the documentation include: cover plates, absorber coatings, thermal insulation, sealants, containment materials, heat transfer fluids, hoses and storage media materials. The primary objectives, scope and principal results of the various studies are presented. The relationship between test results and subsequent consensus standard adoption or revision is drawn where applicable.

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